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Laboratory and Field Evaluations of Methodology for Measuring Emissions of Chlorinated Solvents from Stationary Sources

Entropy Environmentalists, Inc., Research Triangle Park, NC

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LABORATORY AND FIELD EVALUATIONS OF METHODOLOGY FOR MEASURING EMISSIONS OF CHLORINATED SOLVENTS FROM STATIONARY SOURCES

by

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Several chlorinated solvents are being evaluated for future emission regulations by the U.S. Environmental Protection Agency (EPA). Many state and local agencies already regulate chlorinated solvents. Therefore, measurement methodologies for these compounds should be investigated.

Entropy was commissioned by the Quality Assurance Division of EPA's Atmospheric Research and Exposure Assessment Laboratory to perform laboratory and field studies to evaluate sampling and analytical techniques for the measurement of carbon tetrachloride (CCl_4), chloroform ($CHCl_3$), perchloroethylene (PERC), and trichloroethylene (TCE) emissions from stationary sources. The studies were conducted in two phases (1) preliminary work and field studies evaluating EPA Method 18 when applied to the emissions of PERC from degreasing facilities and (2) laboratory and field studies evaluating modified EPA Method 18 sampling procedures for collection of all four chlorinated solvents under high temperature and high moisture conditions.

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FOREWORD

State and local air pollution control agencies presently regulate the emissions of chlorinated solvents. Now the EPA is considering the regulation of the emissions of certain chlorinated solvents. In order to support these regulations, work has been done at a degreasing plant and a high temperature/high moisture facility. Sampling and analysis of perchloroethylene using Method 18 was done at the degreasing plant. However, in the presence of high temperature and high moisture, the current EPA Method 18 does not provide acceptable results. Therefore, further work was done using new techniques applied to EPA Method 18 at a high temperature/high moisture stationary source in which perchloroethylene, trichloroethylene, carbon tetrachloride, vinyl chloride and chloroform were collected and analyzed.

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ABSTRACT

Several chlorinated solvents are being evaluated for future emission regulations by the U. S. Environmental Protection Agency (EPA). Many state and local agencies already regulate chlorinated solvents. Therefore, measurement methodologies for these compounds should be investigated.

Entropy was commissioned by the Quality Assurance Division of EPA's Atmospheric Research and Exposure Assessment Laboratory to perform laboratory and field studies to evaluate sampling and analytical techniques for the measurement of carbon tetrachloride (CCl_4), chloroform ($CHCl_3$), perchloroethylene (PERC), and trichloroethylene (TCE) emissions from stationary sources. The studies were conducted in two phases (1) preliminary work and field studies evaluating EPA Method 18 when applied to the emissions of PERC from degreasing facilities and (2) laboratory and field studies evaluating modified EPA Method 18 sampling procedures for collection of all four chlorinated solvents under high temperature and high moisture conditions.

Based on previous laboratory studies, Method 18 employing Tedlar bag sampling and charcoal adsorption tube sampling techniques was selected for the collection of PERC at degreasing operations. Modifications of Method 18 using in-line gas conditioning systems with Tedlar bags and sorbent tubes were employed for the collection of the solvents under high temperature/high moisture conditions. Sorbent media selected for use after submission to simulated high temperature/high moisture conditions were TRI-X-100^R and Carboxen.^{R} Gas chromatography using flame ionization detection was selected as the analytical technique for Tedlar bag and sorbent tube samples.

Preliminary work for the first evaluation test (1) assessed retention of PERC on charcoal sorbent tubes, (2) identified optimum GC/FID operating parameters for PERC analysis, (3) determined the range and sensitivity of the analytical method, and (4) identified relatively safe desorbing solutions.

A field evaluation of the application of Method 18 to the sampling and analysis of PERC emissions at a degreasing operation was conducted. Eight quadruplicate-train runs were completed on controlled and uncontrolled PERC emissions. The test results indicated that Method 18 Tedlar bag and adsorption tube sampling and GC/FID analysis could successfully be applied to PERC emissions from a degreasing facility. The sampling and analytical precision, in terms of the coefficient of variation, for the evaluation conducted at the degreasing facility was just below 10% for samples collected using Tedlar bags. Linear regression analysis indicated that the standard deviations determined for controlled emissions and uncontrolled emissions were not dependent on emission levels. The average estimated precision in terms of coefficient of variation for the samples collected using charcoal tubes was 4.6% at controlled emissions. No estimate of precision could be made for uncontrolled emissions collection. On-site analytical accuracy, in terms of percent error, determined using audit gas cylinders was 6 to 7%.

In the laboratory evaluations for the second field test, in-line gas conditioning systems were tested by simulating the sampling conditions expected. The evaluation of the adsorption media for use in high temperature/high moisture conditions showed 100% retention for all of the solvents on Carboxen and three of the solvents on TRI-X-100. The desorption efficiency was 82% for all solvents collected on TRI-X-100 when methylene chloride was the desorbing solution. Solvents collected on Carboxen desorbed with methylene chloride yielded a desorption efficiency of 97%.

The second field evaluation was conducted to assess collection of chlorinated solvents under high temperature/high moisture conditions. Five sampling runs (each run collecting 16-20 samples using both Tedlar bags and adsorption tubes) were conducted in a gas-fired boiler stack spiked with known amounts of CCl_4, $CHCl_3$, PERC, and TCE. Chlorinated solvents collected under high temperature/high moisture conditions resulted in sampling and analytical precisions, in terms of the relative standard deviation, of 18.2% for the Tedlar bag samples, 5.85% for the samples collected on TRI-X-100, and 5.17% for the samples collected on Carboxen-564. The method accuracy, in terms of the percent error, determined by spiking the stack with known amounts of the solvents ranged from 8.8 to 9.8%.

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SECTION 1
INTRODUCTION

Several chlorinated solvents are being evaluated by the U. S. Environmental Protection Agency (EPA) for possible future regulations for emissions from stationary sources. Entropy Environmentalists, Inc. (Entropy) assisted the Quality Assurance Division (QAD) of EPA's Atmospheric Research and Exposure Assessment Laboratory (AREAL), Research Triangle Park, North Carolina in evaluation of methods for sampling and analysis of some of the high priority solvents. Those solvents included in the evaluation were carbon tetrachloride (CCl_4), chloroform ($CHCl_3$), perchloroethylene (PERC), and trichloroethylene (TCE).

The work was conducted in two phases: (1) a field evaluation of sampling and analytical techniques for measuring emissions of PERC, and (2) laboratory and field evaluations for the collection of all four solvents under high temperature/high moisture conditions. The field evaluation for the first phase was conducted at a chemical degreasing facility which utilizes PERC. The field evaluation for the second phase was conducted at a gas-fired boiler where known amounts of the four solvents could be introduced into the stack and collected under high temperature/high moisture conditions. The stack temperature was approximately 500°F and the moisture was near 14%.

The use of EPA Method 18[1] was previously evaluated as a sampling and analysis method for PERC emissions in a laboratory study sponsored by the EPA.[2] Since PERC is widely used as a solvent in the chemical degreasing industry, a degreasing facility was selected for field evaluation of the candidate method. Gaseous samples collected in Tedlar bags were analyzed directly using a gas chromatograph equipped with a flame ionization detector (GC/FID). Emission samples were also adsorbed onto activated charcoal in sorbent tubes and likewise analyzed by GC/FID after desorption with an appropriate solvent.

Laboratory evaluation of modified EPA Method 18 techniques included (1) determining the effectiveness of an in-line gas conditioning system for high temperature/high moisture sample collection and (2) the identification of

adsorption media that will effectively collect organic solvents under these conditions. A field evaluation of the sorbents chosen and the gas conditioning system developed was conducted at a gas-fired boiler where the exhaust gas stream was spiked with the chlorinated solvents. Samples collected both in Tedlar bags and on the adsorption tubes were analyzed using GC/FID.

REPORT ORGANIZATION

Section 2 presents the conclusions and recommendations regarding use of Method 18 as a sampling and analysis technique for emissions of chlorinated solvents. Section 3 discusses the procedures and results of the work conducted prior to the first field evaluation test. The procedures and results of the first field test conducted at a degreasing facility are discussed in Section 4. Section 5 presents the procedures and results of the laboratory studies conducted to evaluate collection of the solvents under high temperature/high moisture conditions, and Section 6 discusses the field evaluation test and results for these sampling conditions. Finally, Section 7 presents the quality control and quality assurance procedures implemented for the laboratory and field evaluations.

SECTION 2
CONCLUSIONS AND RECOMMENDATIONS

The applicability of EPA Method 18 to collection of chlorinated solvents at degreasing facilities and facilities with high temperature/high moisture emissions encompasses two phases: (1) sample collection and (2) sample analysis. The precision of the EPA Method 18 sampling and analytical procedures was determined using multiple sampling systems. The accuracy of the application of Method 18 at each type of source was determined by analysis of cylinder audit gases, and at the high temperature/high moisture emission source, by spiking the emissions with a known amount of each solvent. Based on this work, the following conclusions can be made:

- Method 18 sampling procedures and gas chromatography using flame ionization detection (GC/FID) are acceptable for the measurement of PERC emissions from degreasing facilities.
- Based on the multiple train results for samples of controlled and uncontrolled emissions at the degreasing facility, the analytical precision was not dependent upon emission levels.
- The concentration values for individual Tedlar bag samples are unchanged (within $\pm$10%) over time periods ranging from three to five days, if the Tedlar bags do not leak.
- The migration of PERC from the primary to the backup portion of the charcoal tube is insignificant at tube charges from 1.3 to 36.0 ul.
- The most effective desorbing solution for removing PERC from charcoal for GC/FID analysis is methylene chloride.
- Comparison of the data obtained using Tedlar bag and charcoal tube samples collected resulted in a 10% difference with the Tedlar bag sample values being higher than the charcoal tube values at the degreasing facility.
- The precisions, expressed as average coefficients of variation, for the Tedlar bag PERC samples collected at the outlet (controlled emissions) and inlet (uncontrolled emissions) of the degreasing facility were 9.8%

and 9.7%, respectively.

- The precision for the charcoal tube PERC samples collected at the outlet of the degreasing facility was 4.6%.
- The probe length variations examined did not affect the coefficient of variation as compared to unmodified Tedlar bag sampling systems.
- Varying the sampling flow rates did not affect the coefficient of variation as compared to the unmodified sampling.
- Modified Method 18 sampling procedures relying on in-line gas conditioning systems and Method 18 analytical procedures are acceptable for sampling and analysis of carbon tetrachloride (CCl_4), chloroform ($CHCl_3$), perchloroethylene (PERC), and trichloroethylene (TCE) emissions from high temperature/high moisture stationary sources.
- Carboxen-564 is an effective sorbent for sample collection of CCl_4, $CHCl_3$, PERC, and TCE under high temperature/high moisture conditions.
- TRI-X-100 is an effective sorbent for collection of CCl_4, $CHCl_3$, PERC, and TCE under high temperature conditions.
- In-line gas conditioning coupled with Tedlar bag sampling is appropriate for the collection of CCl_4, $CHCl_3$, PERC, and TCE under high temperature/high moisture conditions.
- Comparison of the Tedlar bag and sorbent tube sample data from the high temperature/high moisture source yielded a relative difference of 1.65% with the Tedlar bag sample values being slightly higher than the sorbent tube values.
- The average precision, expressed as the relative standard deviation, for Tedlar bag sampling using an in-line gas conditioning system and GC analysis of chlorinated solvents at a high temperature/high moisture source was 18.2%.
- The average precision for the Carboxen-564 tube samples using a gas conditioning system coupled with GC analysis at the high temperature/high moisture source was 5.17%; for the TRI-X-100 sorbent under similar conditions, it was 5.85% when both the primary and backup tube samples were considered.

Based on the results of this method evaluation, the following recommendations can be made:

- For the measurement of PERC at degreasing facilities, sampling and analysis should be conducted according to the procedures outlined in EPA

Method 18.

- Further investigation is recommended for the possible uses of portable analyzers.
- For measurement of CCl_4, $CHCl_3$, PERC, and TCE emissions from high temperature/high moisture sources, Carboxen-564 coupled with an in-line gas conditioning system may be used for sample collection .
- For Tedlar bag sampling of the same solvents under high temperature/high moisture conditions, an in-line gas conditioning system coupled with Method 18 sampling procedures should be applied.

SECTION 3
PRELIMINARY WORK FOR EVALUATION TEST 1

This section presents the experimental procedures and results of the preliminary work conducted prior to the field test to evaluate Method 18 Tedlar bag and charcoal tube sample collection techniques and GC/FID analysis for measuring PERC emissions from degreasing facilities. The objectives of this preliminary work were:

- o Evaluate the ability of the collection tubes to retain PERC in the primary portion of the tube.
- o Identify effective desorption solvents with the least health and safety risks.
- o Determine the optimum operating parameters for the GC/FID.
- o Determine the range and sensitivity of the Method 18 Tedlar bag and charcoal tube sampling techniques for PERC.
- o Determine the need for modification of the sampling or analytical procedures based on analysis of samples collected during presurvey testing.

EXPERIMENTAL PROCEDURES

A previous EPA study indicated that some chlorinatd solvents are not stable in Mylar bags; therefore, Tedlar bags were used for the study. Double seamed Tedlar bags were fabricated according to a previously described procedure.[2] Sealing male quick-connects were installed on the Tedlar bags in place of Hoke
ball valves so that the Tedlar bag contents could be accessed more conveniently.

Each Tedlar bag was leak-checked before and after being used to collect field samples. Tedlar bags were pressurized to 2 to 4 inches H_2O and allowed to stand for 10 minutes. If a loss of pressure was detected during this period using a differential pressure gauge, the Tedlar bag was discarded and analysis of the Tedlar bag's contents was disregarded.

Charcoal tubes were checked for possible migration of PERC from the

sample tubes. These were prepared simultaneously and charged with various amounts of PERC. The migration of adsorbed PERC from the primary portions of charcoal tubes to the backup portions was examined for tubes stored for 29 days at room temperature.

The major health and safety risk associated with the sampling and analysis of PERC using charcoal adsorption tubes was the use of carbon disulfide to desorb the analyte prior to analysis. An alternative desorption solvent was sought to eliminate the health and safety risks associated with the use of the highly toxic and flammable carbon disulfide. The two alternative solvents investigated were methylene chloride and methanol.

All samples were analyzed using a Perkin-Elmer 8210 gas chromatograph equipped with a flame ionization detector (GC/FID). Columns and operating parameters utilized by others[3] were reviewed to optimize analysis conditions. For all analyses, the hydrogen and air pressures supplied to the FID were maintained at 29 and 22 psi, respectively. These values were selected by optimizing the FID response according to manufacturer's suggestions.

The range and sensitivity of the Method 18 Tedlar bag sampling and charcoal tube sampling and GC/FID analysis techniques were determined. The limit of detection and quantitation were estimated for Tedlar bag and tube samples of PERC as described by Joseph Knoll (QAD) in the Journal of Chromatographic Science.[4]

As specified in Method 18, presurvey samples were collected at the degreasing facility where the first field evaluation would be conducted. This was done to determine any need for modification of the proposed sampling procedures. Gaseous samples were collected in Tedlar bags and charcoal tubes, and quantified by GC/FID according to Method 18. The adsorbed charcoal tube samples were desorbed using a fixed volume of carbon disulfide to allow GC/FID analysis using liquid injection of the resulting solutions. A liquid sample of PERC, steam-desorbed from the carbon bed adsorber, was also collected for GC/FID analysis to identify any interferences that might occur as a result of plant operations during the field test evaluation.

RESULTS

The results of the experiments evaluating migration of PERC from the primary portion of the tube to the backup portion showed an insignificant

portion of the original charge in the backup portion of the tube. The charges to the primary portion of the tube ranged from 1.3 to 36.0 ul. When amounts greater than 5 ul were initially charged, the migration was less than 5% of the charge. The data are presented in Table 1.

TABLE 1. MIGRATION STUDY RESULTS[a]

Tube Portion	Actual Charge (ul)	Amount Detected (ul)	Percent Migration
	Charge range: 1.30 - 5.16 ul		
Backup	0.00	0.18	18%
Primary	1.30	1.01	
Backup	0.00	0.17	6%
Primary	2.68	2.83	
Backup	0.00	0.19	3.7%
Primary	5.16	5.11	
	Charge range: 12.0 - 36.0 ul		
Backup	0.0	0.18	1.9%
Primary	12.0	11.40	
Backup	0.0	0.23	1.2%
Primary	24.0	19.90	
Backup[b]	0.0	0.23	0.6%
Primary	39.1	37.90	

[a]Conditions: Charged to primary portion using syringe. Stored at ambient temperature from October 18 - November 16 (29 days).
[b]Data from single injection rather than average of results from a pair.

In an effort to find solvents less hazardous than carbon disulfide for desorbing the charcoal tubes for GC analysis, methanol and methylene chloride were investigated. Although PERC can be adequately separated chromatographically from either methanol or methylene chloride, the desorption efficiency of methanol for PERC adsorbed on charcoal was considerably below 50%. Methylene chloride, however, can also be separated from PERC using a temperature program, and it exhibited a desorption efficiency of approximately 82% (see Table 2). Based on these data, methylene chloride was chosen for the desorption of the charcoal tube samples in the final field evaluation. However, the combustion products exiting from the GC detector when using this solvent are highly irritating to eyes and lungs, and venting of these exhaust gases is necessary to avoid irritation.

Experiments to identify the optimum operating conditions for the GC/FID analysis of Tedlar bag samples resulted in utilizing a packed chromatography column containing 20% SP-2100/0.1% Carbowax 1500 on 100/120 mesh Supelcoport, operated isothermally at 120°C. Using carbon disulfide as the desorbing solution (presurvey samples), the charcoal tubes were analyzed using a column containing 10% OV-101 on 100/120 mesh Supelcoport, operated isothermally at 110°C. Solutions obtained using methylene chloride as the desorption solvent were analyzed using the OV-101 column and the following temperature programming: 70°C (3 minutes) - ramp of 30°C/min (1 minute) - 100°C (3 minutes). The conditions for analysis of the Tedlar bag samples and the charcoal tube samples collected in the field evaluation are presented in Table 3.

For the Tedlar bag samples, the estimated limit of detection was 1.6 ppm, and the estimated quantifiable limit was 4.8 ppm. No upper detection limit (the point at which the detector becomes saturated and/or the detector response becomes non-linear) was apparent below the level at which room temperature nitrogen is saturated with PERC. For liquid injections of 5.0 ul, dissolved PERC in amounts as low as 0.003 ul per 4 mL of carbon disulfide resulted in a discernable detector response, while concentrations above 160 ul per 4 mL of carbon disulfide saturated the detector.

The levels of PERC measured in the presurvey samples collected at the inlet duct and outlet duct of the carbon bed adsorber on the degreasing facility are presented in Table 4.

TABLE 2. CHARCOAL TUBE DESORPTION COEFFICIENT FOR METHYLENE CHLORIDE

Desorbed Charge(ul)	Calibrated Charge (ul)	Desorption Coefficient
3.45	3.98	0.867
6.43	8.03	0.801
9.42	12.00	0.785
36.50	46.30	0.788
171.00	142.00	0.830
171.00	135.00	0.789
171.00	150.00	0.877

Average value over the entire range: 0.82 +/- 0.04 (95% confidence interval).

TABLE 3. GC CONDITIONS FOR SAMPLE ANALYSIS

Tedlar Bags	
Column	20% SP-2100/0.1%Carbowax on 100/120 mesh Supelcoport (10' x 1/8")
Oven Temperature	120°C (isothermal)
Injector Temperature	200°C
Detector Temperature	300°C
Carrier Flow	30 mL/min
Analysis Time	3.5 min
Charcoal Tubes Desorbed With CS_2	
Column	10% OV-101 on Gas Chrom Q, 100/120 (6' x 1/8")
Oven Temperature	110°C (isothermal)
Injector Temperature	200°C
Detector Temperature	300°C
Carrier Flow	12 mL/min
Analysis Time	3.2 min
Charcoal Tubes Desorbed With CH_2Cl_2	
Column	10% OV-101 on Gas Chrom Q, 100/120 (6' x 1/8")
Oven Temperature	70°C (3 min), 30°C/min (1 min), 100°C (3 min)
Injector Temperature	200°C
Detector Temperature	300°C
Carrier Flow	12 mL/min
Analysis time	7 min

TABLE 4. PRESURVEY RESULTS

	Perchloroethylene Concentration (ppm)		
Sampling Location	Tedlar Bags	Charcoal Tubes	Plant Estimates
Inlet	1139	3600 - 5900	1000-2000
Outlet	trace	not detected	25-50

Two Tedlar bag samples, one each from the inlet and outlet of the carbon bed adsorber, were analyzed along with one charcoal tube sample from each location. The inlet Tedlar bag analysis yielded a PERC concentration of 1139 ppm, while only a trace amount was found in the outlet Tedlar bag sample. Analyses of the charcoal tube samples yielded concentrations of 3000-5000 for the inlet location. No PERC was detected for the outlet location. In the analyses of the presurvey tube samples, PERC was desorbed from the primary portion of each tube with 4 mL of carbon disulfide.

The liquid sample of PERC collected from the carbon bed adsorber yielded spurious chromatographic peaks whose summed integrated areas were less than one percent of the total integrated peak areas. This indicated that the combined impurities in the PERC used by the plant would cause no analytical problems, and no apparent interferences were introduced by the degreasing operation.

SECTION 4
FIELD EVALUATION TEST 1

The first field evaluation test was conducted at a chemical degreasing facility utilizing PERC. The facility employs a carbon bed adsorber to control the PERC emissions.

The primary objective of this field evaluation test was to evaluate Method 18 when applied to sampling and analysis of PERC at a degreasing facility. The technical approach used in the development of the field study was designed to address the following primary objectives:

- o Evaluate the use of Method 18 for sampling and analysis of both controlled and uncontrolled PERC emissions from a degreasing facility.
- o Evaluate the two sampling methodologies described in EPA Method 18: (1) Tedlar bag sampling and (2) adsorption (charcoal) tube sampling for collection of PERC emissions.

A secondary objective of the program was to evaluate a screening technique for PERC emissions using portable VOC analyzers. In addition, the effects of (1) sample line length from the sampling manifold to the individual Tedlar bags and (2) sample flow rate into the Tedlar bags and through the charcoal tubes was briefly examined.

The sampling and analytical field evaluation protocol was finalized based on the results of the preliminary laboratory work and the presurvey results. The test matrix shown in Table 5 was used to collect data to meet the objectives listed above. Specifically, eight dual quadruplicate sampling train (quad-train) runs were conducted at both the inlet and the outlet to the carbon bed adsorber. At each sampling location, one quad-train was used for sample collection in Tedlar bags, and one quad-train was used for sample collection onto charcoal tubes. The quad-trains for the Tedlar bags and charcoal tubes drew gases from a common manifold (see Figure 1).

TABLE 5. TEST MATRIX FOR FIELD EVALUATION TEST 1

Test Day	Location	Morning	Afternoon
1	Inlet	Quad-train 1-I - bags Quad-train 1-I - tubes	Quad-train 2-I,3-I - bags Quad-train 2-I,3-I - tubes
	Outlet	Quad-train 1-0 - bags Quad-train 1-0 - tubes	Quad-train 2-0,3-0 bags Quad-train 2-0,3-0 - tubes
2	Inlet	Quad-train 4-I,5-I - bags Quad-train 4-I,5-I - tubes	Quad-train 6-I,7-I - bags Quad-train 6-I,7-I - tubes
	Outlet	Quad-train 4-0,5-0 - bags Quad-train 4-0,5-0 - tubes	Quad-train 6-0,7-0 - bags Quad-train 6-0,7-0 - tubes
3	Inlet	Quad-train 8-I - bags Quad-train 8-I - tubes	
	Outlet	Quad-train 8-0 - bags Quad-train 8-0 - tubes	

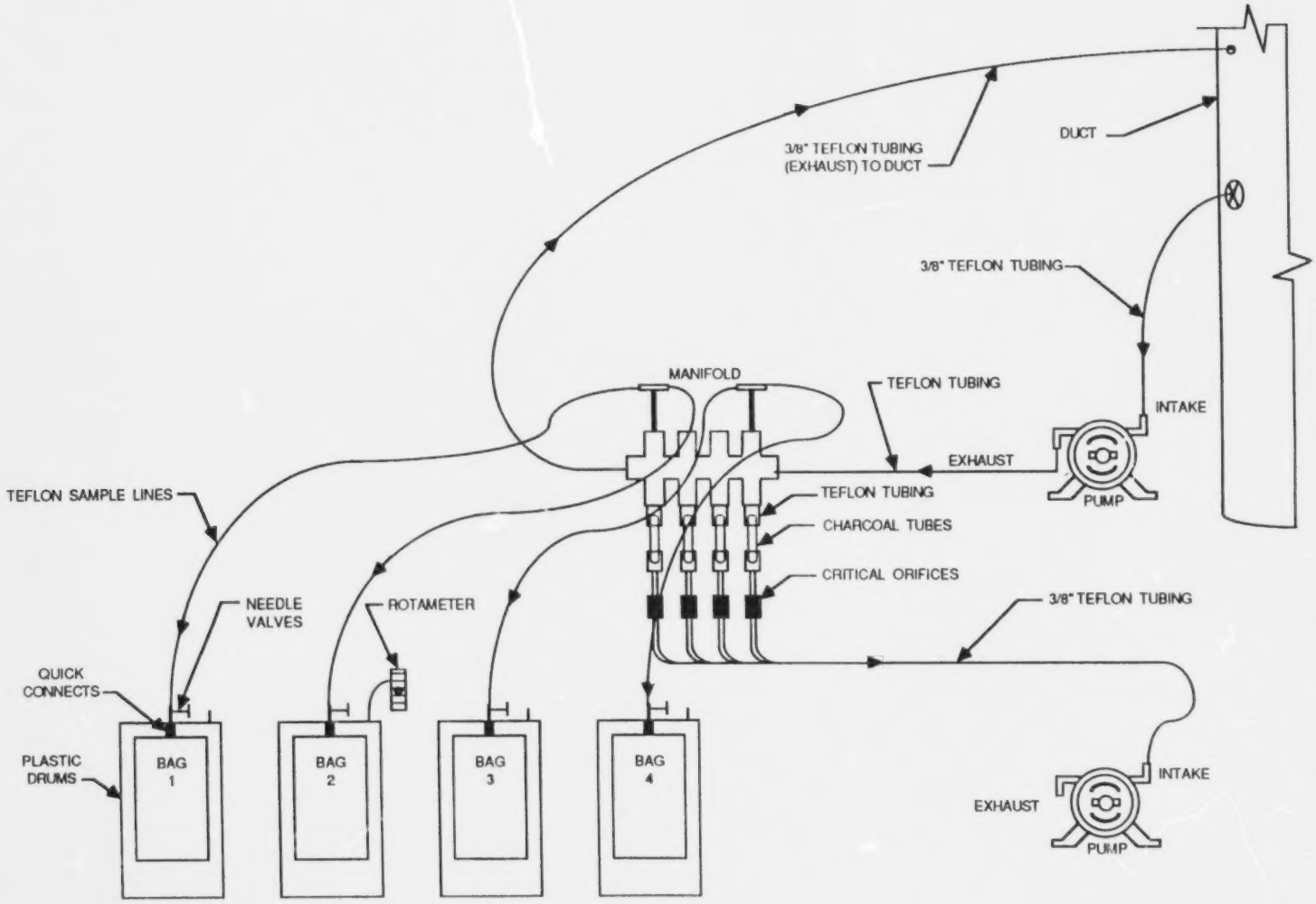

Figure 1. Dual quad-train sampling apparatus.

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EXPERIMENTAL PROCEDURES

Facility Description

The chemical degreasing facility where the first field evaluation test was conducted employs a dual carbon bed adsorber for controlling PERC emissions. A schematic of the system is presented in Figure 2. While one carbon bed is in the adsorption mode, the other is in the regeneration mode. General operating procedures at this facility result in PERC emissions of approximately 50 ppm at the adsorber outlet, and 1000 to 2000 ppm at the adsorber inlet.

Sampling Location

Gas samples were acquired at the outlet (representing controlled emissions) and the inlet (representing uncontrolled emissions) of the dual carbon bed adsorber. The location of sampling ports relative to the carbon bed adsorber is shown in Figure 2. The sampling location had sufficient space to accommodate the test personnel and equipment required for the field evaluation.

Sampling Procedures and Sampling Equipment

All sampling was conducted according to EPA Method 18. Sampling runs were conducted for an hour. Much of the evaluation paralleled a previous field evaluation concerning the sampling and analysis of trichloroethylene and ethylene dichloride emissions.[5]

A schematic of the dual quadruplicate-train (quad-train) sampling system is presented in Figure 1. Separate probe lines constructed of 3/8" Teflon tubing were inserted into both the inlet and outlet ducts of the carbon bed adsorber. Each probe was connected to a Teflon-diaphragm sampling pump. The output of each pump was conducted via Teflon tubing to individual manifolds constructed of stainless steel piping. The exhaust from these manifolds was returned via Teflon tubing to the appropriate duct. Before each sampling run, the manifolds were purged with stack gas for 5 minutes to allow equilibration of the system.

Tedlar bag samples were simultaneously collected from each steel manifold using individual Teflon sample lines (approximately 6 feet in length) to direct the sample gas, driven by the positive pressure present in the manifolds, to a set of four Tedlar bags at the inlet and a set of four Tedlar bags at the outlet (see Figure 1). Each of the eight Tedlar bags (four connected to each manifold) was enclosed in a PVC drum (see Figure 3). A female bulkhead quick-connect, affixed in each drum lid, was attached to each

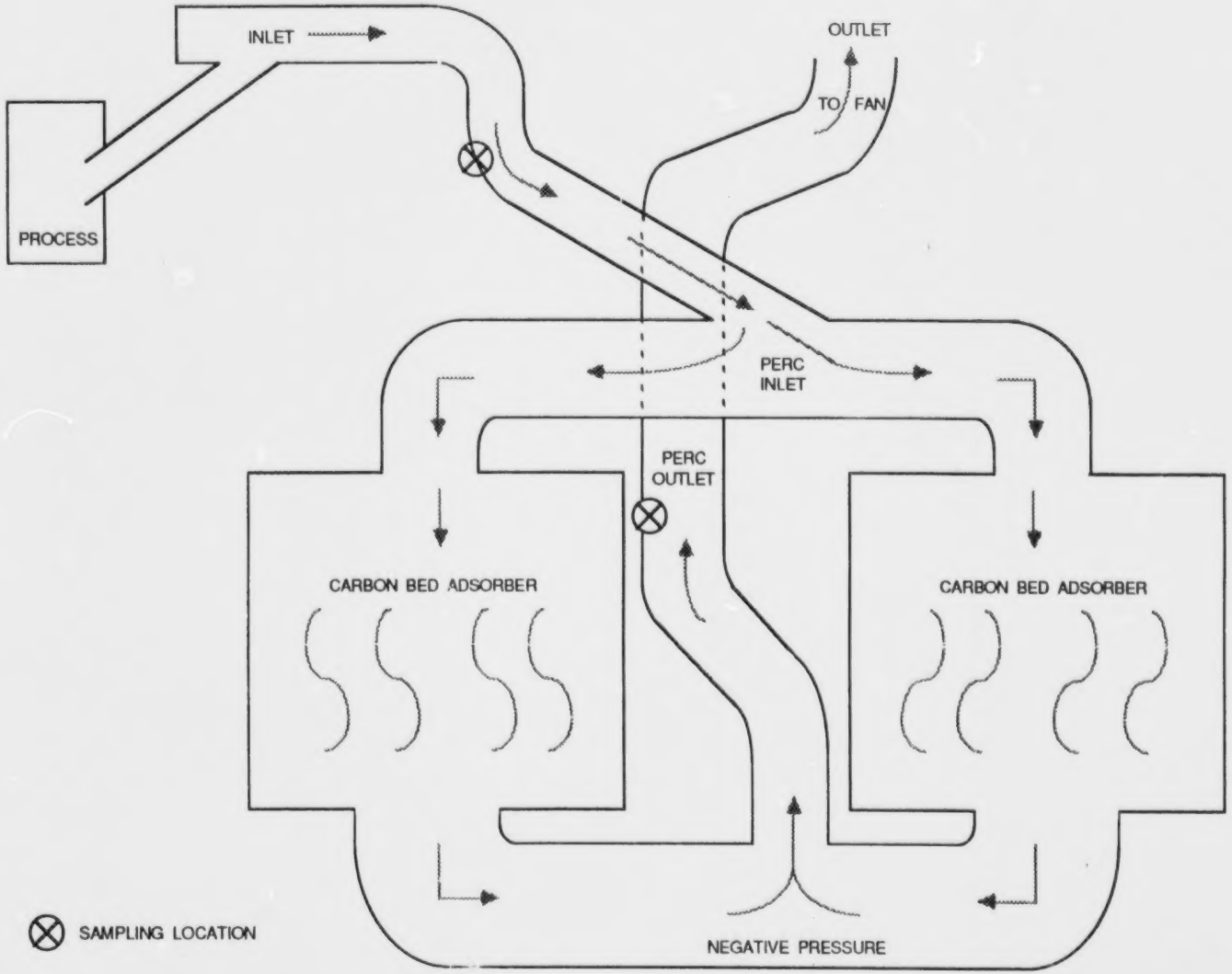

Figure 2. Dual carbon bed adsorber.

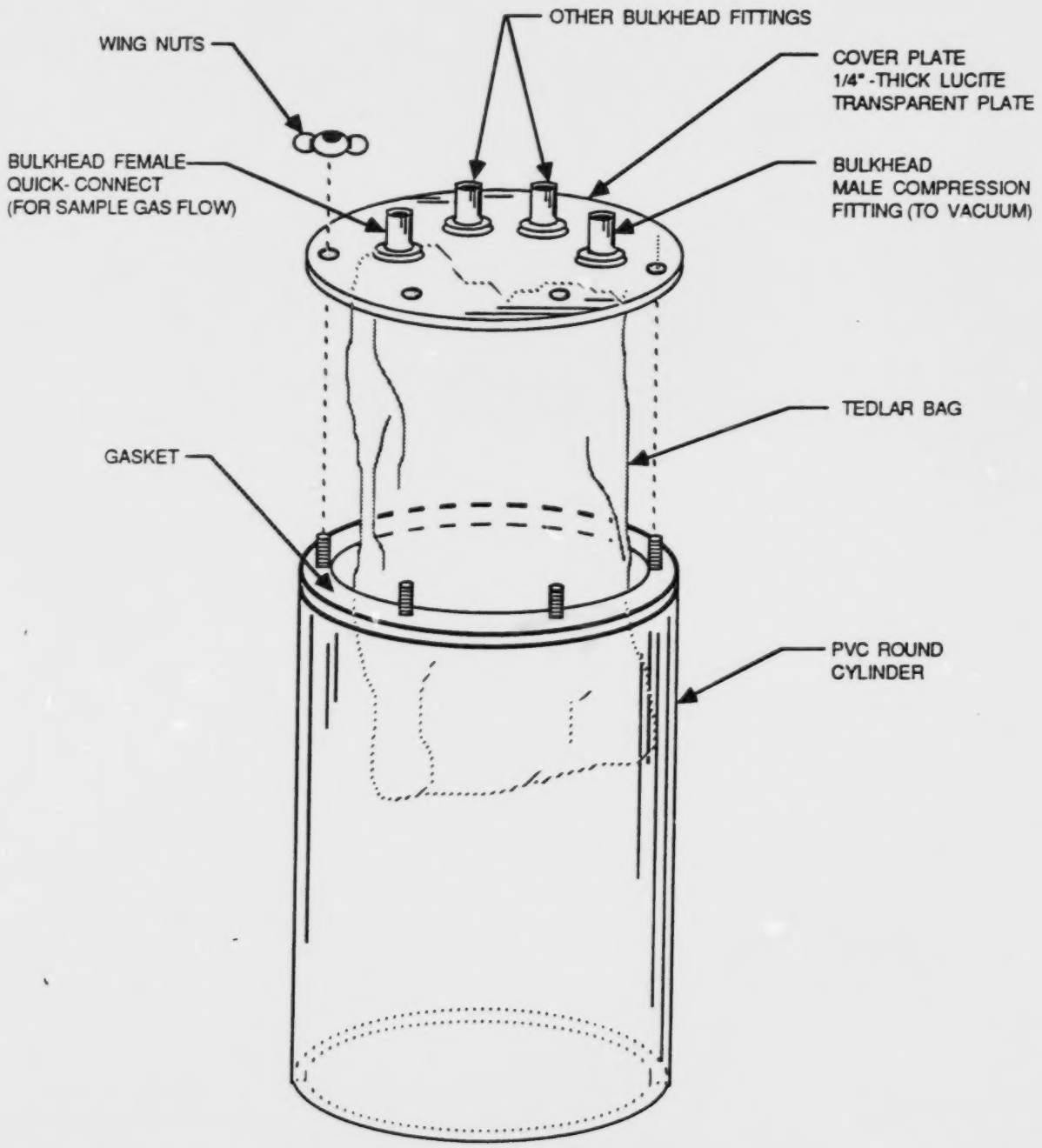

Figure 3. PVC bag sampler.

sample line. Tedlar bags were attached via their male connects to the underside of the drum lids before the drums were sealed.

Flow into each Tedlar bag was moderated by individual, adjustable needle valves and monitored by measuring the air flow displaced from each drum using a rotameter. Flow into the Tedlar bags was maintained at 55 - 60 cm^3/min at the outlet and 40 - 45 cm^3/min at the inlet.

Charcoal tube samples were collected simultaneously with the Tedlar bag samples at the outlet and inlet using quad-trains. Four tubes of activated charcoal (SKC 226-16) were attached to the steel manifolds by short pieces of Teflon tubing (see Figure 1). Flow through each tube was controlled using a calibrated critical orifice and a sufficiently high pump vacuum (in this case, 15 in. Hg) behind each orifice so that sonic velocity was maintained. Each orifice was calibrated before and after field use. If these calibration values differed by less than 5%, their average was used in calculating flow rates during sampling. If the two values differed by a larger amount, the samples collected using that orifice were not considered in the data analysis.

To assess the effect of sample line length from the sampling manifold to the individual Tedlar bag samples, the tubing used to connect the individual Tedlar bag sampling containers to the common manifold was lengthened from 6 to 9 feet for two members of the Tedlar bag quad-train on the sixth run. The effect of varying sample flow rate within a quad-train was also examined to ascertain if increased variability in replicate analyses occurred. The flow on the final run was modified from 55 - 60 cm^3/min to 30 cm^3/min for two of the outlet Tedlar bag samples collected, and from 40 - 45 cm^3/min to 25 cm^3/min for two of the inlet Tedlar bag samples. Also on the final run, flow through two of the charcoal tubes at the outlet to the carbon bed adsorber was increased from 1.3 L/min to 6.0 L/min.

A secondary objective of the field test was to evaluate a screening technique for PERC emissions. PERC emissions from the inlet and outlet of the carbon bed adsorber were introduced into two portable analyzers by attaching their probes to the appropriate sampling manifold. The analyzers used were a Bacharach^R TLV Sniffer and an HNU^R Model ISPI 101 Intrinsically Safe Portable Photoionization Analyzer. An AID^R Model 712 Portable Total Hydrocarbon Analyzer was also to be evaluated, but when a zero background reading could not be obtained, use of the instrument was abandoned.

The TLV Sniffer has a range of 1 to 10,000 ppm for combustible gases and vapors. To detect and measure concentrations of combustible gases in the air, the TLV catalytically oxidizes the gas in a pumped-in sample of air by means of a catalyst-coated resistance element. Oxidation of gas produces changes in the element's resistance which are proportional to the amount of gas oxidized. These changes in resistance are converted to an electrical signal which corresponds to the concentration of gas present.

The Photoionization Analyzer equipped with a 10.2 eV lamp has a sensitivity range of 1 to 2,000 ppm for a variety of ionizable organic gases. The photoionization process involves passing the sample gas through a chamber with a sealed UV light source which ionizes the organic molecules; this ionization is measured and converted into an analog readout of the concentration.

To calibrate the portable monitors in the field, cylinders of PERC in air were purchased. This custom blend contained a concentration of 50 ppm PERC. Manufacturer's analysis of the cylinder was certified to $\pm$ 2%. During the quad-train sample runs, readings from the analyzers were taken every five minutes. The individual readings were taken as representative of the emission level during the previous 5-minute interval. These readings were used in calculation of an average emission level over the entire period used for collection of Tedlar bag and charcoal samples (usually 60 minutes). Each portable analyzer was alternately located at the inlet and at the outlet manifold so that each was used for a number of measurements at both locations.

Analytical Procedures and Equipment

All field samples collected were analyzed by Entropy using a Perkin-Elmer 8210 gas chromatograph equipped with a flame ionization detector. Peak areas were determined using an electronic integrator. Instrument conditions for analysis were specified in Table 3.

Tedlar Bag Samples --

Analysis of Tedlar bag samples was conducted on-site utilizing a 0.1 mL sample loop in conjunction with a manually-activated gas sampling valve. Samples were drawn into the loop by a small pump. See Table 3 for instrument conditions.

Preparation of calibration standards for the GC analysis of PERC in the laboratory and the field was modified to eliminate the use of the midget impinger, heated by boiling water, required by the procedure outlined in Method 18. An alternate setup, shown in Figure 4, was utilized. Samples were prepared by passing nitrogen gas (A) through an electrically heated injection port (D) into Tedlar bags (G) after metering through a mass flow analyzer (C). Known volumes of PERC were injected into the stream via the port during filling of the Tedlar bags.

The mass flow analyzer was calibrated at a fixed flow rate using a bubble flow meter (H) attached after the injection port (not heated during calibration) at the three-way valve (E). The standard volume of gas, in liters, passing first through the analyzer, and then to the injection port and bubble meter, was calculated using the observed displacement in the bubble meter and the observed ambient temperature and pressure. The formula used was:

$$V_{std} = \frac{(0.3857)(P_{amb})}{(T_{amb} + 273.16)} (V_{BM}) \qquad \text{Equation 1}$$

where:

V_{std} = Standard volume of gas, liters,
P_{amb} = Barometric pressure, mm Hg,
T_{amb} = Ambient temperature, °C, and
V_{BM} = Measured bubble meter volume, liters.

The pressure at the outlet of the gas regulator (B) was maintained at a constant value of 20 psi during both the calibration and preparation of standard gas mixtures. The nominal flow rate (uncorrected meter value) was also fixed, this at 800 ml/min.

A calibration factor y for use of the meter during preparation of standards was obtained by dividing the metered volume into the calculated standard volume used for calibration. This calculated factor y was used in determining the PERC concentrations, in ppm, of prepared standards from the volumes of liquid PERC and nitrogen gas introduced into the Tedlar bag:

$$\text{Concentration} = \frac{V_{liq}}{y \times V_{N_2}} \times 235.38 \qquad \text{Equation 2}$$

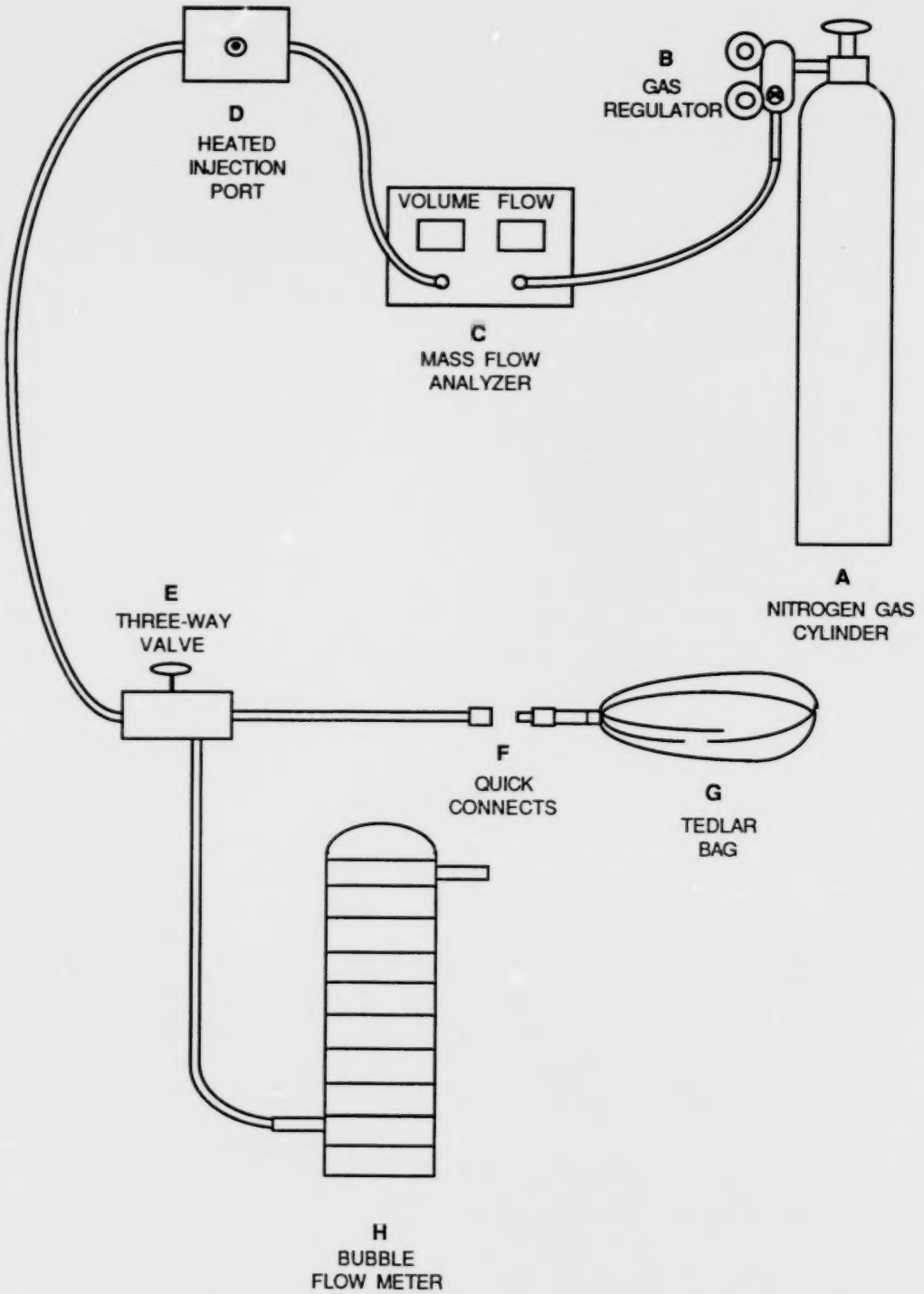

Figure 4. Apparatus for preparing Tedlar bag calibration standards.

where:

V_{liq} = Volume of liquid PERC injection, ul,
y = Calibration factor of mass flow meter, unitless, and
V_{N_2} = Volume of metered N_2, liters.

The conversion factor 235.38 (ppm - L/ul) corrects for the density of PERC at 20°C, the molecular weight of PERC, the standard molar volume of an ideal gas, and the change to ppm units.

To prepare calibration standards in the field, liquid PERC was injected directly into Tedlar bags fitted with septa using 10 and 100 ul gastight syringes as Tedlar bags were filled with a known volume of nitrogen. For low concentration standards, PERC was diluted first in methylene chloride to one part in eleven by volume. For the lowest concentration standard, 9.85 ppm, an NBS standard reference cylinder was used.

Calibration data were collected for two ranges. A least squares linear regression line was calculated for instrument response in two ranges: 10 - 100 ppm and 100 - 10,000 ppm. Each calibration was audited using cylinders of PERC in nitrogen provided by the Quality Assurance Division of EPA, Research Triangle Park, North Carolina.

Charcoal Tube Samples --

Charcoal tube samples were maintained at or near 0°C until analysis could be completed at the Entropy base laboratory. All tubes were analyzed within twelve days of collection, using 4 mL of methylene chloride to desorb the PERC from the primary portion of each tube. The backup portions of representative tubes were checked for possible breakthrough (displacement of the analyte from the primary portion of the adsorption tube due to the flow of gas through the tube). Backups were also analyzed for the tubes showing the largest PERC charges to determine if the adsorbent's full capacity was ever exceeded.

Standards for charcoal tubes were prepared in 4 ml of methylene chloride. For the calibration standards and most of the samples, 5 ul were injected into the GC. For unusually dilute or concentrated samples, the injection size was modified to bring detector response near the calibration range.

The formula used to convert the amount of PERC present on a tube to the corresponding gas concentration, in ppm, flowing through the tube is given below:

$$\text{Concentration} = \frac{V_P}{(DE)(V_s)} \times 235.38 \qquad \text{Equation 3}$$

where:

V_p = Volume of PERC on tube, ul,
DE = Desorption efficiency, unitless, and
V_s = Volume of sample gas passed through tube, liters.

The conversion factor 235.38 corrects for the density of PERC at 20°C, the molecular weight of PERC, the standard molar volume of an ideal gas, and the change to ppm units. The volume of PERC on a tube was determined using standard solutions and was corrected for losses arising from the adsorption/desorption process, by introduction of the unitless factor DE. The sampling time was recorded for each run while the flow through each charcoal tube was fixed using critical orifices.

Data Analysis

Pooled standard deviations were sought for the analytical results of the Tedlar bag and charcoal tube samples. A statistical comparison of the values obtained by the two methods was conducted.

For both the inlet and the outlet Tedlar bag sample PERC values, linear regression of the populations' means on their standard deviations indicated that the standard deviations determined at the two locations were not level dependent. Bartlett's test for homogeneity of population variance indicated that for both the inlet and outlet values, the calculation of a pooled standard deviation was inappropriate. Instead, the coefficients of variation of individual sample runs were averaged for the inlet and outlet sampling locations to provide a crude estimate of overall precision.

Although linear regression analysis indicated a slight level dependence of the standard deviations observed for the outlet charcoal tube sample PERC values, the result of a Bartlett's test showed that the variances for this population were essentially homogeneous. A pooled standard deviation was calculated for the charcoal tube samples.

Linear regression of the mean PERC values obtained using charcoal tubes on the mean PERC values obtained using Tedlar bag for selected outlet sampling runs was performed. No attempt was made to correlate results from the Tedlar bag and charcoal tube analyses of inlet samples since only single, rather than replicate, values were available for the charcoal tube samples from this location.

RESULTS

All four bags from each quad-train run were initially analyzed on-site by Entropy. For the Tedlar bag samples collected, the successive duplicate analyses specified by Method 18 were generally well within 5% of their mean values as required by the method. In fact, the duplicate analyses often differed from the mean by less than 1%. The average coefficient of variation observed for the outlet Tedlar bag samples was 9.8%, while a value of 9.7% was observed for the inlet samples. A summary of the results is presented in Table 6.

The on-site analysis of cylinder audit gases provided by the Quality Assurance Division, Research Triangle Park, North Carolina, allowed estimation of method accuracy. The audit performed in the 10 - 100 ppm range yielded a result that was 6.0% of the certified value. In the range of 100 - 1000 ppm, the audit value differed from the certified value by 7.0%.

To assess the stability of PERC in the Tedlar bags and the within laboratory precision, Entropy analyzed selected Tedlar bag samples upon return to the base laboratory. One Tedlar bag from each run, and all the Tedlar bags from the last day's run were retained for reanalysis. As shown in Table 7, Tedlar bags which did not leak-check after analysis generally yielded much lower concentration values when reanalyzed in the laboratory. For almost all the Tedlar bags that passed the leak-check, the concentration values were unchanged (within $\pm$ 10%) over time periods ranging from 3 to 5 days. This difference underscores the importance of a post-run leak-check of all Tedlar bags.

For the charcoal tube analysis, a summary of the results is presented in Table 8. Successive duplicate analyses of desorption solutions were generally within 1 to 3% of their mean values. A pooled standard deviation of 14 ppm was calculated for charcoal tube samples collected at the outlet. The average coefficient of variation for these samples was 4.6%. Unfortunately, flows through three of the four orifices used to collect samples at the inlet location were found to have changed significantly from their original calibrated rates. No estimate of the precision could be calculated because only one reliable measurement was obtained from each run using this methodology.

Comparison of Tedlar bag and charcoal tube sample data from the outlet showed a difference of 10% (using the slope of the regression line as a crude

TABLE 6. SUMMARY OF ANALYTICAL RESULTS AND PRECISION DATA FOR TEDLAR BAG SAMPLES

Date	Run ID[a]	Mean Concentration (ppm)	Std. Dev. (ppm)	Coef. of Var.[b] (%)
		Outlet Samples		
12/2	1-0	613.0	134.0	21.9
12/2	2-0	210.0	16.0	7.8
12/2	3-0	43.5	4.0	9.3
12/3	4-0	196.0	4.0	2.0
12/3	5-0	605.0	100.0	16.6
12/3	6-0	69.8	4.6	6.7
12/3	7-0	284.0	26.0	9.0
12/4	8-0	988.0	50.0	5.1
		Inlet Samples		
12/2	1-I	9510	1300	13.6
12/2	2-I	9780	100	1.0
12/2	3-I	1980	400	20.0
12/3	4-I	7970	190	2.4
12/3	5-I	10,500	120	1.1
12/3	6-I	6310	1160	18.4
12/3	7-I	6590	450	6.8
12/4	8-I	9450	1350	14.3

aO = Outlet, I = Inlet.

bCoefficient of Variation = [Std. Dev./ Mean] x 100%.

TABLE 7. STABILITY OF TEDLAR BAG SAMPLES: COMPARISON OF FIELD AND LABORATORY ANALYSES

Sample ID*	Field Conc.(ppm)	Lab Conc.(ppm)	Average Conc.(ppm)	Age (days)	Percent Change
Tedlar bags that passed post-analysis leak-check					
AO-2	537	506	522	5	-5.8%
CO-4	212	194	203	5	-8.5%
EO-3	49	33	41	5	-32.7%
JO-3	556	519	538	4	-6.7%
LO-3	68	69	69	4	1.5%
QO-2	1122	1042	1082	3	-7.1%
BI-4	10624	10158	10391	5	-4.4%
KI-4	10415	10218	10317	4	-1.9%
PI-3	6158	5552	5855	4	-9.8%
RI-1	9953	9850	9902	3	-1.0%
RI-4	8622	4167	6395	3	-51.7%
Tedlar bags that did not pass post-analysis leak-check					
GO-2	201	187	194	4	-7.0%
NO-4	298	228	263	4	-23.5%
QO-3	1018	746	882	3	-26.7%
DI-3	9886	9952	9919	5	-0.7%
FI-1	2278	329	1304	5	-85.6%
HI-3	7937	6526	7232	4	-17.8%
MI-4	7842	6943	7393	4	-11.5%

*Position on Sampling Manifold (e.g., A)/Sampling Location (e.g., O) - Run Number (e.g., -2).

TABLE 8. SUMMARY OF ANALYTICAL RESULTS AND PRECISION DATA FOR CHARCOAL TUBE SAMPLES

Date	Run ID	Mean Concentration (ppm)	Std. Dev. (ppm)	Coef. of Var.[a] (%)
		Outlet Samples		
12/2	A	526.0	26.0	4.9
12/2	C	164.0	8.0	5.0
12/2	E	44.0	1.8	4.2
12/3	G	173.0	4.0	2.0
12/3	J	542.0	18.0	3.3
12/3	L	252.0	16.0	6.5
12/3	N	241.0	7.0	3.0
		Inlet Samples		
12/2	B	12,100		
12/2	D	10,800		
12/2	F	2720		
12/3	H	8700		
12/3	K	11,100		
12/3	M	5340		
12/3	P	7190		
12/4	R	8780		

aCoefficient of Variation = [Std. Dev./ Mean] x 100%.

estimate). The values obtained using the charcoal tubes averaged slightly less than the values obtained for the Tedlar bag samples.

For the brief examination of the effects of varied sample line length and Tedlar bag sampling flow rates, the samples collected with the probe modifications (extended 3 feet) yielded coefficients of variation within the range of the values obtained for the unmodified sample runs. The results obtained for both the probe length modification (6-O and 6-I) and the sample flow rate modification (8-O and 8-I) are included in Table 6. It appears from these results that probe length is not a factor in sampling. Flow through two of the charcoal tubes was increased from 1.3 L/min to about 6.0 L/min (sample run 8-O). However, no conclusion could be drawn concerning the effect of this change, because the backup portions of all members of this quad-train showed PERC levels exceeding 10% of that collected on the primary portion of the tube.

Data sheets for each test day's calibration, audits, and the field and laboratory analysis of Tedlar bag and charcoal tube samples are included as Appendix A to this document.

For the screening technique, each portable analyzer was alternately located at the inlet and outlet. Comparison of the emission levels determined using the analyzers with those determined using GC analysis of the Tedlar bag and the charcoal tube samples reveals that agreement is erratic. The portable analyzers, charcoal adsorption tube data, and Tedlar bag data are presented together in Table 9. The results indicate that PERC concentrations measured at the outlet using the HNU photoionization analyzer are in fair agreement with measurements obtained upon analysis of Tedlar bag and charcoal samples. A parallel statement can be made about the performance of the Bacharach TLV Sniffer at the inlet, where the concentrations measured were approximately 2,000 to 10,000 ppm. Additional evaluations of these devices are warranted.

TABLE 9. SUMMARY OF ANALYSES

Run ID	Average Perchloroethylene Concentration (ppm)			
	Tedlar Bags	Charcoal Tubes	TLV	HNU
Outlet				
A	613.0	526.0	99	---
C	210.0	164.0	---	70
E	43.5	44.0	---	54
G	196.0	173.0	40	---
J	605.0	542.0	---	700
L	69.8	252.0	0	---
N	284.0	241.0	---	0
Q	988.0	UNAVBL	---	1,030
Inlet				
B	9,510	12,100	---	1,750
D	9,780	10,800	9,800	---
E	1,980	2,720	2,020	---
H	7,970	8,700	---	1,510
K	10,500	11,000	9,350	---
M	6,310	5,340	---	6,300
P	6,590	7,190	*	---
R	9,450	8,780	8,290	---

*An "average" was not calculated because battery failure prevented completion of the sample run.

SECTION 5
LABORATORY EVALUATION FOR TEST 2

This section presents the laboratory evaluation carried out prior to the field test evaluating application of EPA Method 18 to measure emissions of chlorinated organics under high temperature and high moisture conditions. The chlorinated solvents used in the laboratory and field evaluations were: carbon tetrachloride (CCl_4), chloroform ($CHCl_3$), perchloroethylene (PERC), and trichloroethylene (TCE).

The specific objectives of the laboratory evaluation were to:

- Identify adsorption media that can effectively collect solvents under high temperature/high moisture conditions.
- Determine the best desorbing solution for the selected adsorption media.
- Develop an in-line gas conditioning system to cool the gas stream and eliminate collection of moisture during Tedlar bag sampling.
- Develop an appropriate system for spiking an emission source with known amounts of the chlorinated solvents.
- Determine the optimum operating parameters for the GC/FID analyses.
- Determine any need for modification of the sampling or analytical techniques based on the presurvey test results.

EXPERIMENTAL PROCEDURES

Method 18 sampling at high temperature/high moisture sources has not previously been investigated by the EPA. Necessary modifications to the method included a product search for a sorbent capable of collecting chlorinated organics under high temperature/high moisture conditions and development of a system to allow Tedlar bag sampling under the same conditions.

Based on the manufacturer's specifications, seven sorbent products were selected for laboratory evaluation. These are described in Table 10. All sorbents were pretreated according to manufacturer's instructions. The

TABLE 10. SORBENTS INVESTIGATED FOR USE UNDER HIGH TEMPERATURE/HIGH MOISTURE CONDITIONS

Manufacturer	Sorbent	Description	Common Use
Bio-Rad	Bio-Beads SM-2	Nonpolar polystyrene adsorbent Surface area 300 m^2/g	Concentrate organic material prior to GC analysis
Bio-Rad	Bio-Beads SM-4	Nonpolar polystyrene adsorbent Surface area 725 m^2/g	Concentrate organic material prior to GC analysis
Supelco	Amberlite XAD-8	Intermediate polarity polymeric resin	Sample clean-up prior to chromatographic analysis
Supelco	Carbotrap	Graphitized carbon	Airborne contaminates
Supelco	Carboxen	Hydrophobic carbon molecular sieve	Replaces charcoal for collection of airborne contaminates
Waters	C_{18}-Octadecyl	Highly hydrophobic adsorbent	Water sampling analysis
Union Carbide	TRI-X-100	Inorganic highly hydrophobic silica oxide	Industrial adsorption for waste removal

sorbents were packed into sampling tubes (similar to the charcoal tube) and subjected to retention and breakthrough tests.

Each sorbent was first tested for it's ability to retain the solvents under ambient conditions. Sorbent tubes were challenged with a mixture of CCl_4, $CHCl_3$, PERC, and TCE at 100 ppm each from a Tedlar bag at an approximate flow of 1.0 L/min for a 1-hour period using a diaphragm pump. The gas exiting the tube was introduced directly into the GC/FID. If none of the solvents were detected in the gas exiting the tube for a specific media, further evaluations were conducted, including its performance under high temperature/high moisture conditions.

High temperature/high moisture stack gas containing the chlorinated solvents was simulated by drawing the same mixture of gas at the same flow for the same time period through a heated impinger containing 400 ml of water. The temperature at the exit of the impinger was approximately 170°F. As shown in Figure 5, the stack gas was then drawn through a condenser as it would be in the field, where it was cooled, yielding a condenser exit temperature of approximately 60°F. The gas then was passed through the tube containing the sorbent under investigation, followed by a knock-out jar and a backup sorbent tube. The pressure differential of the system was monitored, as was the gas flow rate. The resulting sample gas was introduced directly into the GC/FID allowing real-time determination of the time at which the media reached breakthrough (no longer retaining one or more of the solvents).

Upon determination of the best sorbents for retaining the chlorinated solvents under high temperature/high moisture conditions, the efficiency with which the solvents could be removed from the sorbent for analysis (desorption efficiency) was investigated. Desorption efficiency for two of the seven sorbents was determined for three different desorbing solutions: acetonitrile, methanol, and methylene chloride. Tubes of each sorbent were directly charged with 1.0 ul of each of the chlorinated solvents. A separate tube was used for each desorbing solution and set aside for one hour prior to use. Using a separate tube for each sorbent/desorbing solution combination, the tubes were desorbed with 4 ml of the appropriate solution. The percent recovery of each solvent from the sorbent was calculated based on the known charge of that solvent on the tube. The desorbing solution yielding the highest recovery for each chlorinated solvent was chosen for use in the field evaluation.

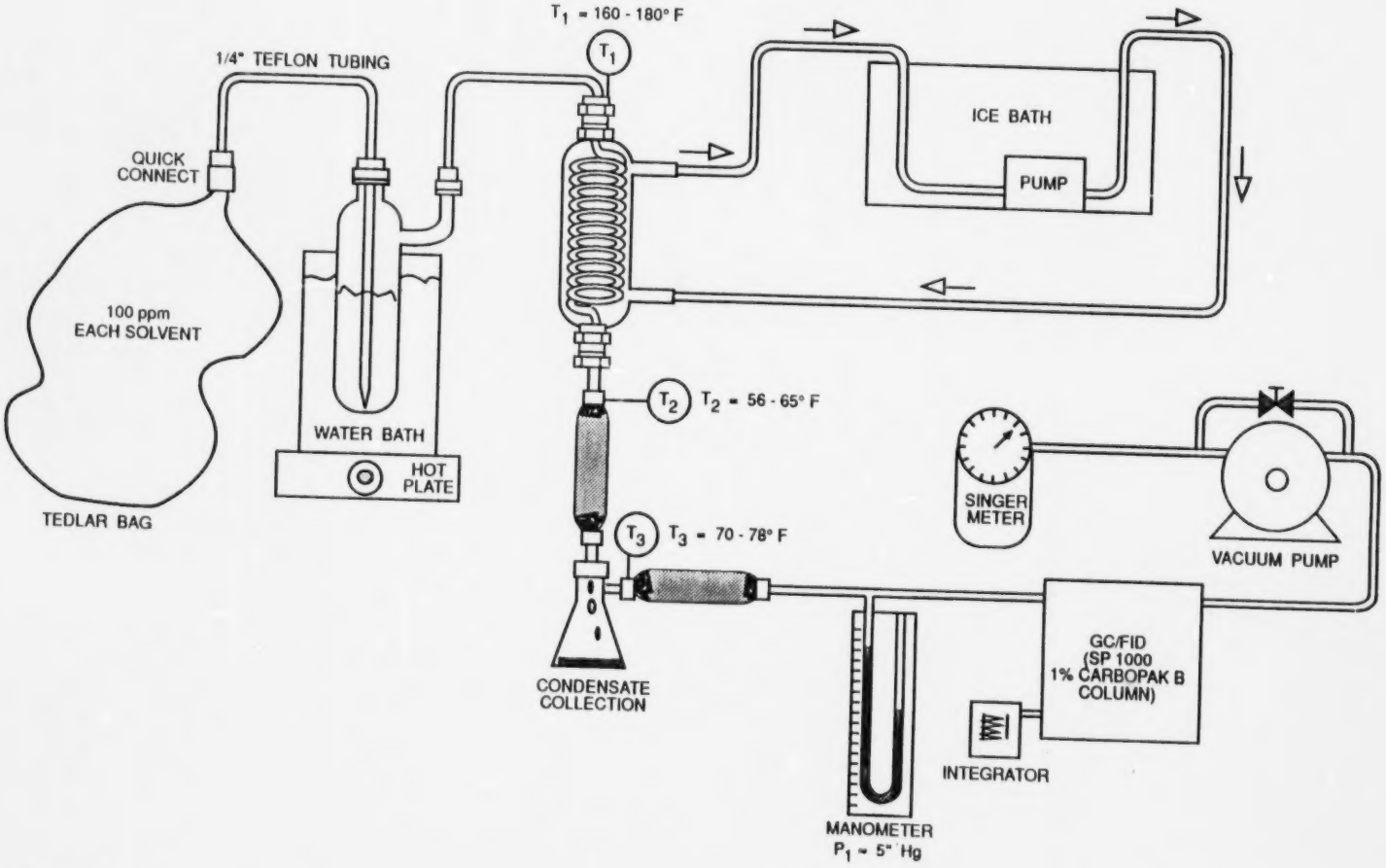

Figure 5. Adsorption breakthrough system.

In-Line Gas Conditioning System for Tedlar Bag Sampling

To apply Tedlar bag sampling to a high temperature/high moisture source, an in-line gas conditioning system was designed. The system consisted of a condenser to cool the gas stream and a knock-out jar to collect the moisture. The Tedlar bag was then placed in-line behind the knock-out jar. A detailed schematic of the system is presented in Figure 6. The water collected in the knock-out jars was analyzed by passing the condensate over a C_{18} cartridge, eluting the cartridge with methanol, and injecting this solution into the GC/FID, or by placing the condensate in 4 ml of methylene chloride following the sample run and injecting this solution.

Development of Emission Spiking System

The inability to locate a high temperature/high moisture stationary source with emissions of chlorinated solvents under investigation resulted in the decision to select a local test site with relatively clean emissions which could be spiked with known amounts of these solvents. A mixture of the solvents equivalent to the desired stack gas concentrations at the sampling point needed to be passed through a pressurized atomizer to create a fine mist that would be vaporized in the high temperature stack. To accomplish this, a Speedaire^{R} paint sprayer was modified to include a deVilbus nebulizing nozzle. This system allowed pressurization of the mixture of solvents through the atomizer to create a fine mist. System pressure was used to regulate the solvent flow. A Teflon flow meter was also placed in line for fine adjustment of the flow rate. A schematic of the spiking system designed is presented in Figure 7. To ensure the flow was regulated at the desired rate, a known amount of H_2O (200 ml) was sprayed through the system at three nominal rates (30, 60, and 120 mL/min) in the range of the anticipated field test rates. The mist from the nozzle was collected in a graduated cylinder, and the flow rate was measured.

Presurvey Sampling

An initial visit to the plant hosting the high temperature/high moisture field evaluation test enabled the design of the spiking and sampling system to be tailored to the specific site. A preliminary test run was conducted to determine any need for modification in the sampling systems. Gaseous samples

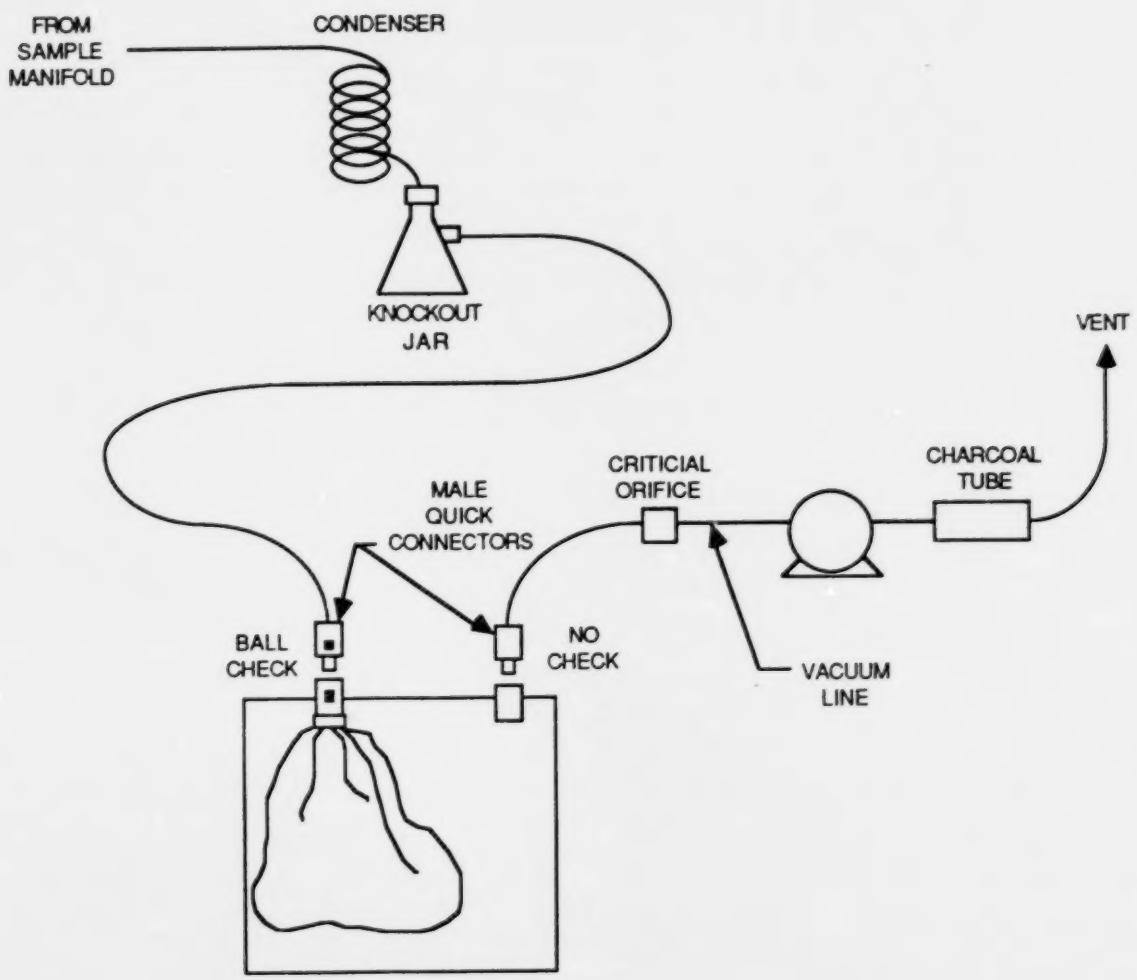

Figure 6. Detailed schematic of Tedlar bag sampling systems.

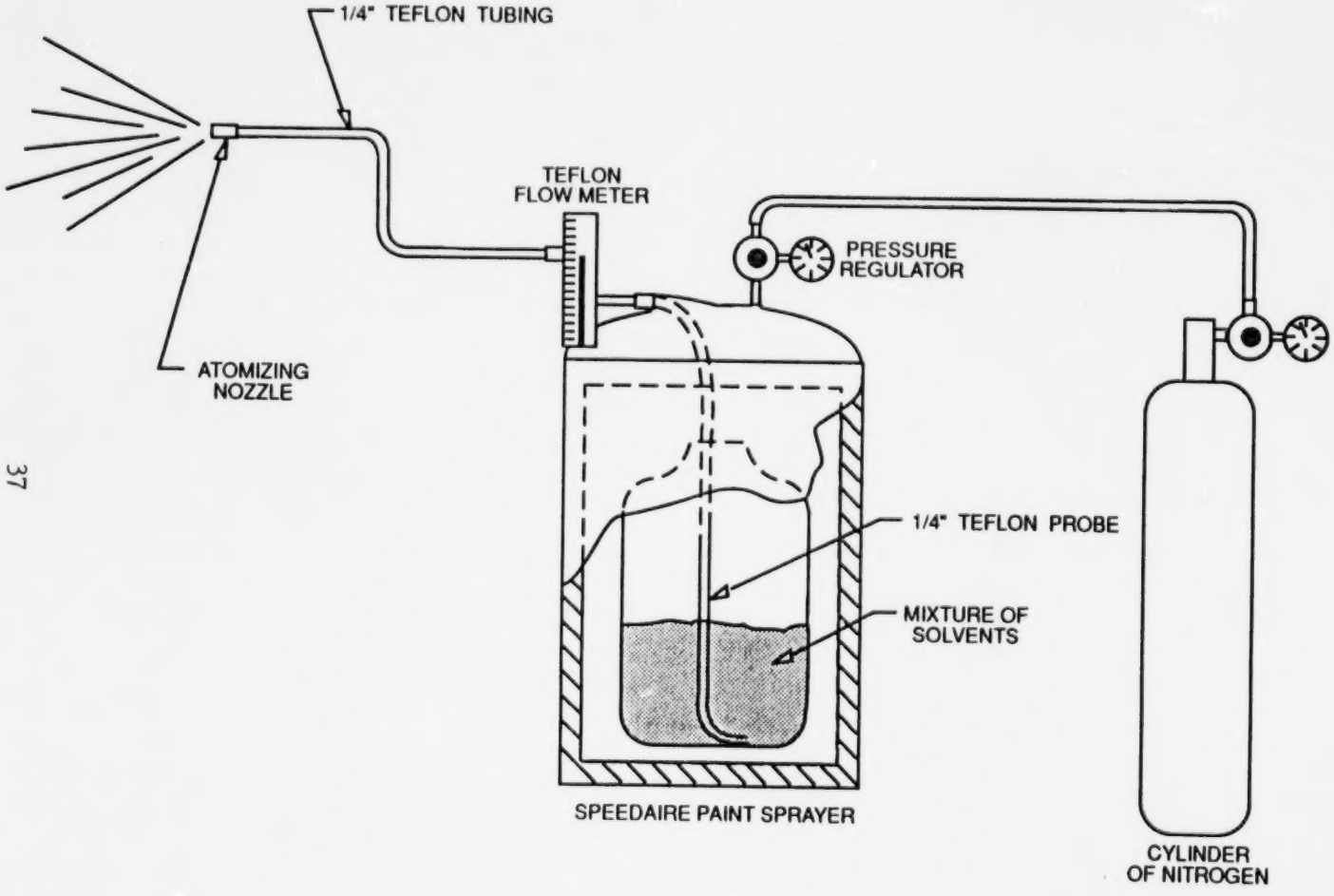

Figure 7. Solvent mixture spiking system.

were collected in four Tedlar bags and quantified by GC/FID. Twelve sorbent tube samples were collected, desorbed using 4 mL of methylene chloride, and quantified by GC/FID.

Sample Analysis

All samples were analyzed by Entropy personnel using a Hewlett-Packard 5890A series gas chromatograph equipped with dual flame ionization detectors (GC/FID). One of the injection ports was equipped with a manually operated gas sampling valve while the other was equipped for liquid injections. Column selection and operating parameters utilized by others[3] were reviewed before final analysis conditions were selected. For all analyses, the hydrogen and air pressures supplied to the dual FID's were maintained at 22 and 20 psi, respectively. These values were selected by optimizing the FID response according to manufacturer's suggestions.

RESULTS

Based on the laboratory breakthrough studies, two of the seven sorbents listed in Table 10, Carboxen and TRI-X-100, were considered acceptable for collection of the solvents under high temperature/high moisture conditions. The results are presented in Table 11. The Carboxen-564 showed no breakthrough for any of the solvents after 158 minutes of sampling. The TRI-X-100 showed 20% breakthrough of CCl_4 after 63 minutes, while all other solvents were adsorbed by the sorbent. These two sorbents were therefore chosen for field evaluation. The TRI-X-100, however, had to be particle-sized before use in the sorbent tubes to obtain more activated surface area. This was accomplished by reducing the particles to 30 mesh size and washing the residual fines from the sorbent with deionized water.

To determine the best desorbing solution for Carboxen and TRI-X-100, acetonitrile, methanol, and methylene chloride were evaluated. The desorption efficiencies obtained for each are presented in Table 12. Although each of the chlorinated solvents were resolved from each of the desorbing solutions, methylene chloride proved to be the most efficient in removing the solvents from both sorbents, with the lowest recovery being 82%. The average recovery for methanol was 70%; however, for removing $CHCl_3$ from the TRI-X-100, and PERC from the Carboxen-564, the percent recovery was zero.

TABLE 11. RESULTS OF LABORATORY BREAKTHROUGH STUDY OF SORBENTS UNDER HIGH TEMPERATURE/HIGH MOISTURE CONDITIONS

Sorbent	Moisture (%)	Average Stack Temperature (°F)	Flow (L/min)	Run Time (min)	Total Gas Volume (L)	Breakthrough (%)
TRI-X-100	46	155	0.590	63	39.66	0-$CHCl_3$ 20-CCl_4 0-PERC 0-TCE
Carboxen	65	173	0.460	158	73.11	0-All Solvents
Bio Beads SM-2 & SM-4	*	Product showed 80% breakthrough after 2 minutes and was eliminated from further evaluation.				
Amberlite XAD-8	*	Product showed 50% breakthrough after 5 minutes and was eliminated from further evaluation.				
Waters C_{18}	*	Product showed 100% breakthrough after 6 minutes and was eliminated from further evaluation.				
Carbotrap	*	Product showed 100% breakthrough after 60 seconds and was eliminated from further evaluation.				

TABLE 12. DESORPTION EFFICIENCIES FOR TRI-X-100 AND CARBOXEN USING VARIOUS DESORBING SOLUTIONS

Desorbing Solution	% Recovery, $CHCl_3$	% Recovery, CCl_4	% Recovery, PERC	% Recovery TCE
TRI-X-100				
Methanol	0	89	50	98
Methylene Chloride	103	82	120	94
Acetonitrile	93.6	91.3	98.7	86.7
Carbonex-564				
Methanol	74	99	0	11.2
Methylene Chloride	112	117	97.2	108
Acetonitrile	79.5	78.4	17.6	48.7

Acetonitrile yielded favorable results for the majority of the solvent/sorbent combinations; however, its products of combustion during analysis using flame ionization are more hazardous than those from methylene chloride. Methylene chloride was therefore chosen for desorption of the sorbent tubes for the remaining GC analyses.

The in-line gas conditioning system developed to eliminate moisture collection in the Tedlar bags has been described above. In the laboratory, the condensate collected was analyzed to confirm that the solvents were not being collected by passing it through an Analytichem-C_{18} cartridge to remove the solvents, eluting them with methanol, and injecting this solution into the GC/FID. None of the solvents were detected.

In evaluating the accuracy of flow rates for the spiking system at the levels measured manually (30 mL/min, 60 mL/min, and 120 mL/min), the flow rate was found to be accurate using water as the atomized solution.

Experiments conducted to identify the optimum instrument operating conditions resulted in analysis of both the Tedlar bag samples and the adsorption tube samples using a packed chromatographic column of 1 % SP-1000 on 60/80 Carbopack-B Supelcoport, operated isothermally at 200°C. The carrier gas (nitrogen) flow rate was maintained at 20 mL/min for both the liquid and the gaseous sample injections.

The analysis of the samples collected during the preliminary test resulted in an average relative standard deviation of 14.1% and an average accuracy of ±9.6% for the solvents collected on TRI-X-100. The average relative standard deviation for the solvents collected on the Carboxen-564 was 10.0%, with an average accuracy of ±8.8%. For the Tedlar bag sample analysis, the precision in terms of the average relative standard deviation was 6.1%, with an average accuracy of ±9.8%. All adsorption tubes were desorbed with 4 mL of methylene chloride and the resulting solutions were analyzed using GC/FID.

The only problem noted with the sampling system was that the sample conditioning devices connected to the end of the manifold farthest from the stack gas flow were not collecting as much condensate as those at the opposite end. As a result, the sorbent tubes were not equally exposed to the high moisture conditions and system modification was necessary. The manifold was wrapped with a heating coil to prevent loss of heat at the end of the manifold. Also discovered in the preliminary test run, was the clogging of some of the

critical orifices. This was most likely caused by fine particles of the sorbent moving through the sorbent tubes and into the sampling system. The sampling system was modified to include filters in-line behind the sorbent tubes.

SECTION 6
FIELD EVALUATION TEST 2

The test site selected for the second field evaluation test was a gas-fired boiler at a local pharmaceutical manufacturing plant. The boiler provided the high temperature and high moisture conditions for sampling and was easily spiked with the four organic solvents of interest, $CHCl_3$, CCl_4, PERC, and TCE.

The primary objective of this field test was to evaluate the application of Method 18 sampling procedures under high temperature and high moisture conditions. Specifically, the field test was designed to:

- Evaluate adsorption media which efficiently collect $CHCl_3$, CCl_4, PERC, and TCE, but pass water vapor, and
- Evaluate the use of an in-line gas conditioning system in conjunction with Method 18 Tedlar bag sampling.

The final field protocol and test matrix (see Table 13) were based on the results from the laboratory evaluation and the preliminary test run described in Section 5. Specifically, five multiple-train sampling runs were conducted, with each resulting in 10 or more samples for GC/FID analysis.

The effect of a modified flow rate on the Method 18 Tedlar bag sampling procedure was also examined. Method 18 recommends a sampling rate of 0.5 L/min, which, for a one-hour sample run requires a Tedlar bag size of approximately 50 L. In a previous study sponsored by the EPA,[6] a flow rate of 0.050 L/min was found to be acceptable for Method 18 Tedlar bag sampling of butadiene, which requires a Tedlar bag size of only 5 L. Therefore, 12 of the 16 Tedlar bag samples collected for the second field test were collected at this lower flow rate using smaller Tedlar bags. To demonstrate that the modified flow rate did not affect sampling, four Tedlar bag samples were collected at the recommended Method 18 flow rate using 50 L Tedlar bags.

TABLE 13. TEST MATRIX FOR SECOND FIELD TEST

Test Day	Test Run AM	Test Run PM	Solvent Concentration
1	Run 1*	Run 2	Run 1 ≥5 ppm Run 2 ~25 ppm
	4 small bags** 4 TRI-X-100[R] tubes 4 Carboxen[R] tubes	4 small bags 4 large bags*** 4 TRI-X-100 tubes 4 Carboxen tubes	
2	Run 3	Run 4	Run 3 ~25 ppm Run 4 ~55 ppm
	4 small bags 4 large bags 4 TRI-X-100 tubes 6 Carboxen tubes	4 small bags 4 Carboxen tubes 6 TRI-X-100 tubes	
3	Run 5		Run 5 ~25 ppm
	6 TRI-X-100 tubes 4 Carboxen tubes		

*Preliminary run.
**Tedlar bags, approximately 5 L.
***Tedlar bags, approximately 50 L.

Facility Description

To quantify the method accuracy, to allow simultaneous measurement of the four solvents of interest under high temperature/high moisture conditions, and to eliminate out-of-town travel, a local source with relatively clean high temperature/high moisture emissions which could be spiked was sought. A gas-fired boiler at a local pharmaceutical manufacturing plant was the source chosen for the field evaluation.

Sampling Location

Sampling was conducted in the 2-foot, 3-inch diameter exhaust stack at the Unit No. 1 boiler of the facility. The 3-inch sampling port was approximately 15 feet above the breeching inlet to the stack, 3 feet above the roof of the boiler house, and 12 feet below the top of the stack. The sample gases were drawn from a single point near the center of the stack into a sampling manifold. The boiler which is rated at 15,000 pounds per hour of steam, was operated at a constant full load during the testing and discharged about 200,000 scfh at 600°F, 14% moisture, and 7% excess air. The solvent spray was introduced into a port in the breeching just prior to the stack.

Sampling Procedures and Sampling Equipment

Much of this field evaluation test paralleled the field test conducted to evaluate the sampling and analysis of PERC at a degreasing facility. Sampling runs were conducted according to EPA Method 18 with minor modifications for the high temperature/high moisture sampling conditions. Sampling runs were conducted over a one-hour time period.

Schematics of the complete sampling system are presented in Figures 8 and 9. The probe, which was constructed of 3/8-inch Teflon tubing, was inserted into the port with the inlet at the center of the stack approximately 18 feet from the point of injection of the solvents into the stack. A diaphragm pump was used to pull the gases into a heated sampling manifold which was constructed of stainless steel piping. Twenty sampling lines were attached to the manifold, allowing generation of up to 20 simultaneous samples from a single sampling run. Eight of the sample lines were equipped for Tedlar bag sampling, while the other twelve were equipped for sorbent tube sampling. The exhaust from the manifold was returned to the stack via Teflon tubing. Both

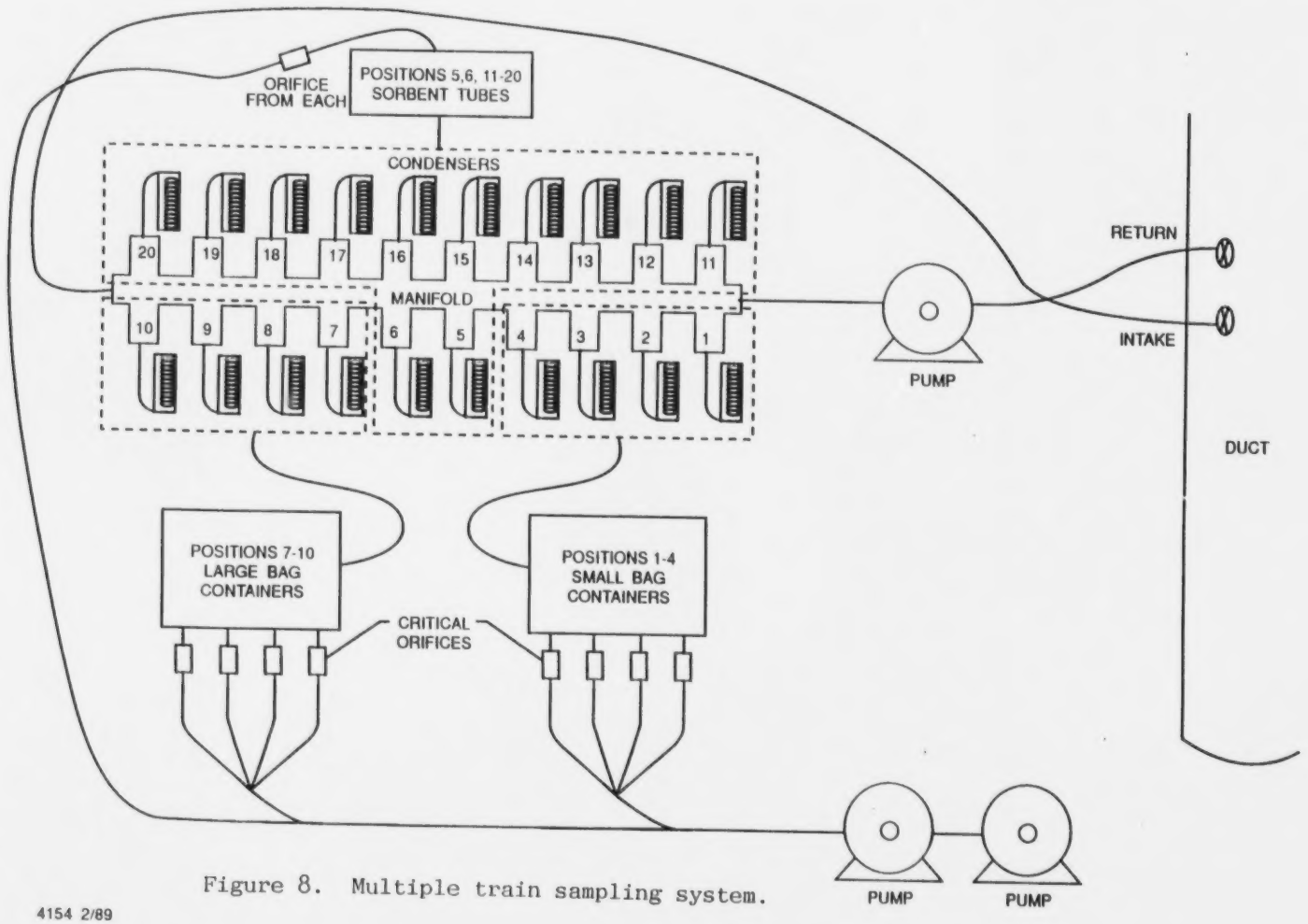

Figure 8. Multiple train sampling system.

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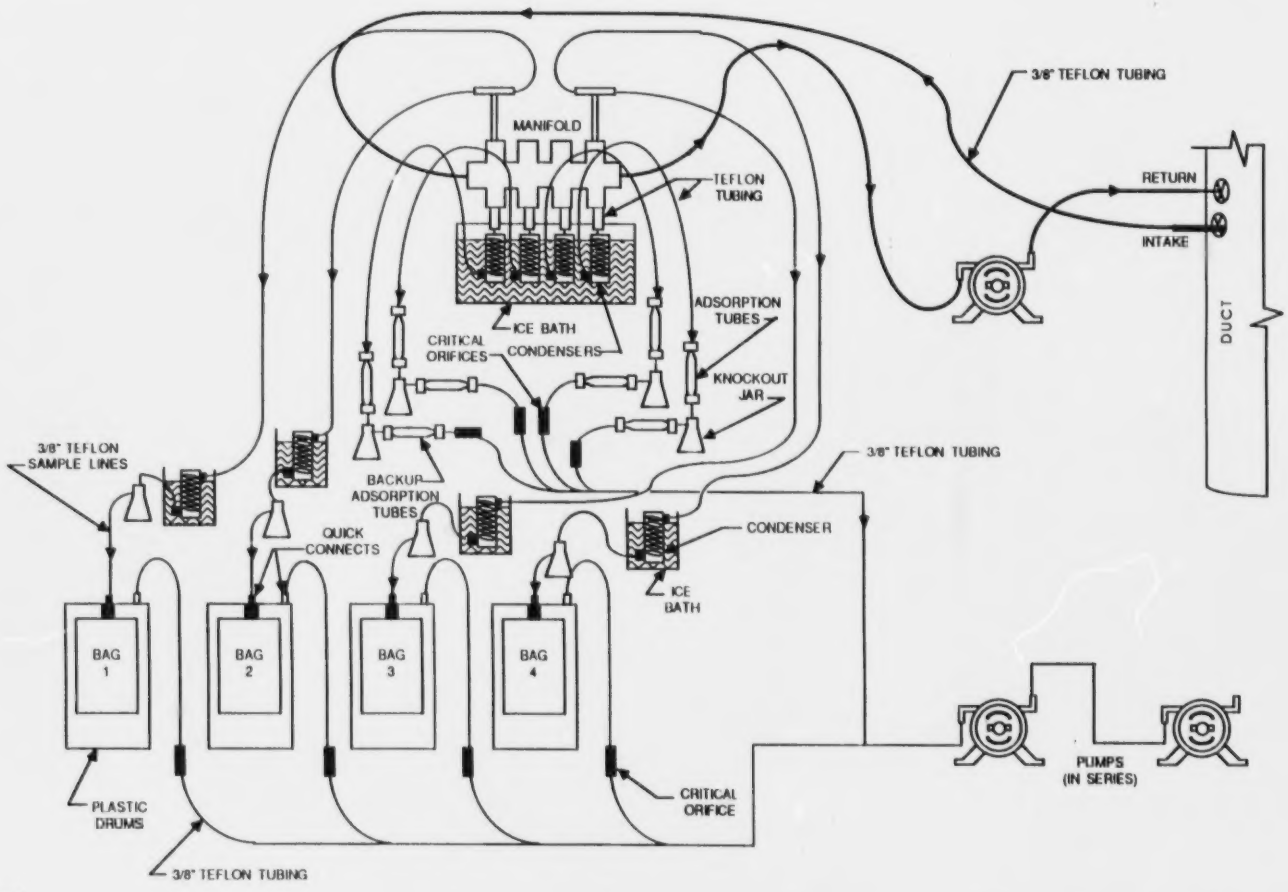

Figure 9. Detailed schematic of multiple train sampling system.

the Tedlar bag and sorbent tube samples were collected from the manifold using two Thomas® diaphragm pumps in series.

Each sample line from the manifold was constructed of 10 feet of 1/4-inch Teflon tubing wrapped in a spiral to create a condenser, which was submerged in an ice water bath to cool the gas. Following the condenser section, the Teflon tubing was connected either (1) to a knock-out jar for collection of the condensates and then the Tedlar bag for Tedlar bag sampling or (2) to a sorbent tube, then to a knockout jar and to a backup tube for tube sampling (see Figures 6, 9, and 10).

The Tedlar bags were enclosed in rigid PVC drums (refer to the first PERC field test for specifications). A male quick-connect was attached to the end of each Tedlar bag sample line which was approximately six feet in length. The sample line was attached to the Tedlar bag by the quick-connect on the lid of the PVC drum holding the Tedlar bag. A sample was drawn into the Tedlar bag by creating a vacuum inside the rigid container. The sample flows into the Tedlar bags were controlled by individual calibrated critical orifices. A detailed schematic of a Tedlar bag sampling system is presented in Figure 6.

As previously mentioned, a sampling rate less than that specified by Method 18 was used for 12 of the 16 Tedlar bag samples collected. To validate the use of the reduced flow rate, one set of four Tedlar bag samples was collected at the higher sampling rate. The actual flow rates for the small Tedlar bag samples were between 0.038 and 0.040 L/min, while the higher flow rates were 0.440 to 0.470 L/min.

The TRI-X-100 and the Carboxen-564 sorbent tube samples were also collected from the manifold. A short piece of Teflon tubing connected each condenser to a primary sorbent tube. A knock-out jar was placed in line behind the primary tube via Teflon tubing and a secondary (backup) tube followed. A detailed drawing of the sorbent tube sampling system is presented in Figure 10. Flow through each set of tubes (primary and backup) was controlled using calibrated critical orifices and a sufficiently high pump vacuum (17 in. Hg) so that sonic velocity was maintained. The flow rate through the twelve individual tube sets was between 0.230 and 0.240 L/min.

The system developed for spiking the stack with equal amounts of a known concentration of each of the solvents is described in Section 5. The amount of each solvent required to reach the desired stack gas concentration of

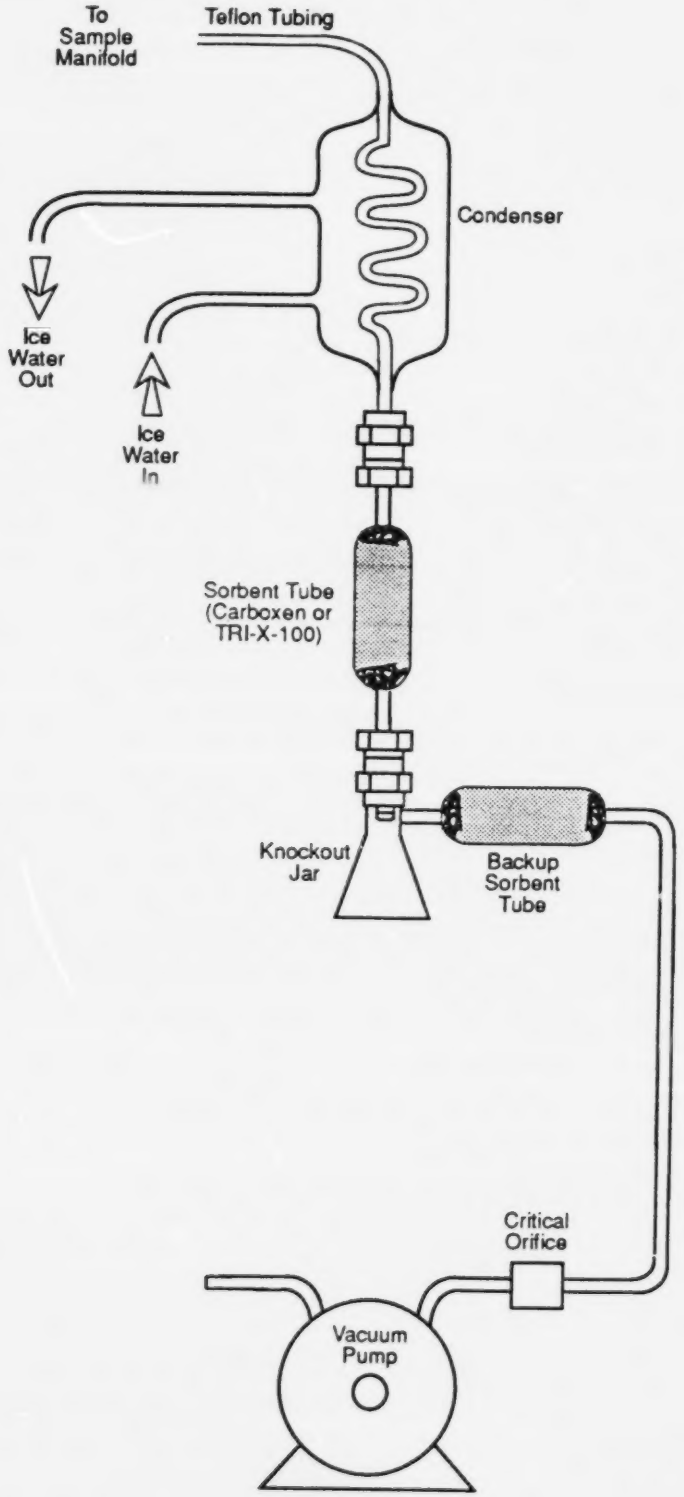

Figure 10. Detailed schematic of sorbent tube sampling system.

approximately 25 parts per million (ppm) was calculated based on the stack diameter, stack velocity, and the composition of each solvent. The mist from the spiking system was introduced into the stack through a single port approximately 18 feet upstream from the sampling port.

Each sampling run was conducted over approximately a 1-hour period. The spiking system was started 5 minutes before the sampling was initiated to allow the solvent mist to vaporize and reach the sampling port. The flow rate of the solvent mixture was monitored and the run continued until all of the solvent mixture was introduced into the stack. Sampling then continued for 5 minutes after all the solvent mixture had been injected into the gas stream.

All samples, both Tedlar bag and sorbent tube, were returned to the Entropy laboratory for GC/FID analysis. The Tedlar bag samples were analyzed within 4 hours of sampling, and the sorbent tube samples were analyzed within 5 days of sampling.

Analytical Procedures and Equipment

All field samples collected were analyzed using a Hewlett-Packard 5890A series gas chromatograph equipped with dual flame ionization detectors. Peak areas were determined using an electronic integrator. Instrument conditions for analysis were presented in Section 5.

Tedlar Bag Samples--

Analysis of the Tedlar bag samples was conducted utilizing a 0.1 mL sample loop in conjunction with a manually-activated gas sampling valve. Samples were drawn into the loop via a small pump.

Standards for Tedlar bag samples were prepared according to the procedures described in Section 4. Calibration data were collected for two ranges: 10 - 50 ppm and 20 - 100 ppm. A least squares linear regression line was calculated for instrument response in those ranges. Each calibration was audited using audit cylinders of each of the solvent provided by the Quality Assurance Division (QAD) of the EPA's Atmospheric Research and Exposure Assessment Laboratory.

Sorbent Tubes--

Sorbent tube samples were maintained at or near 0°C until analysis could be completed. All tubes were analyzed within 5 days of sample collection using methylene chloride to desorb the solvents from the sorbent media. The primary and backup tubes were analyzed using the instrument conditions described in Section 4.

Standards for the adsorption tubes were prepared in 4 mL of methylene chloride. For calibration standard and sample analysis, injections of 0.5 ul were used. The formula used to convert the amount of solvent on a tube to the corresponding gas concentration flowing through the tube is given below:

Equation 4

$$\text{Concentration (ppm)} = \frac{V_{sol}}{[DE][V_{gas}]} \times \frac{d}{MW} \times M_{IG_v} \times 1000$$

where:

V_{sol} = Calculated volume of solvent on tube, ul,
DE = Desorption efficiency coefficient, unitless,
V_{gas} = Volume of sample gas passed through the tube, liters,
d = Density of solvent, g/ml,
MW = Molecular weight of solvent, g/mole,
M_{IG_v} = Molar ideal gas volume (24.056 l/mole).

The amount of solvent on a tube was determined using standard solutions and was corrected for losses arising from the adsorption/desorption process.

Data Analysis

To assess the precisions for the Tedlar bag and sorbent tube sampling and analytical techniques, pooled standard deviations were sought for the multiple sampling train results for the Tedlar bag and sorbent tubes. A comparison of the values obtained by the two methods was conducted.

For both the Tedlar bag and the sorbent tube samples, linear regression of the populations' means was conducted. Bartlett's test for homogeneity of population variance indicated that for all values the calculation of pooled standard deviation was appropriate. Therefore, a pooled standard deviation was calculated for Tedlar bag samples and adsorption tube samples.

RESULTS

All Tedlar bag samples from each run were analyzed within 5 hours of sampling. For the Tedlar bag samples collected, the successive duplicate analyses specified by Method 18 were generally well within 5% of their mean values as required by the method. The overall average relative standard deviation (RSD) for the small Tedlar bag samples was 13.5%, and the overall average RSD for the large Tedlar bag samples was 23%. The results from each run are presented in Tables 14 through 18.

The analysis of cylinder audit gases provided by QAD allowed estimation of method accuracy. The audits performed resulted in the following percent accuracies: CCl_4 - 9.3, $CHCl_3$ - 20.6, PERC - 10.9, and TCE - 7.5.

For the sorbent tube analysis, a summary of the results from each run is also presented in Tables 14 through 18. Successive duplicate analyses of desorption solutions were generally within 5% of their mean values. Based on Bartlett's test for homogeneity of variance, a pooled standard deviation was calculated for both the TRI-X-100 sorbent tubes and the Carboxen sorbent tubes. These calculations include the primary and secondary tube catches for each tube. For Carboxen, over 90% of the solvents were collected on the primary tube, while the TRI-X-100 tubes exhibited as much as 50% breakthrough to the secondary tube when the sorbent was exposed to high moisture conditions. The pooled standard deviations for TRI-X-100 tubes were: 4.28% for CCl_4, 12.95% for $CHCl_3$, 3.13% for PERC, and 3.06% for TCE. The calculated pooled standard deviations for Carboxen were: 6.09% for CCl_4, 5.92% for $CHCl_3$, 5.50% for PERC, and 3.18% for TCE. The average standard deviation for solvent collection on TRI-X-100 was 5.85%, while the average standard deviation for collection on Carboxen was 5.17% using a combination of the primary and secondary tube values. Data sheets for all analyses are contained in Appendix A.

TABLE 14. RUN 1 TEDLAR BAG AND TUBE RESULTS FOR SPIKED ORGANIC COMPOUNDS

	Chlorinated Compound (ppm)			
	$CHCl_3$	CCl_4	TCE	PERC
Run 1 Calculated In-Stack Concentration	28.5	24.0	25.6	22.7
Small Tedlar Bags				
SB-1-1[a]	26.5	20.6	19.4	17.5
SB-2-1	29.2	22.1	21.2	20.5
SB-4-1	29.0	23.6	26.4	18.6
AVERAGE	28.3	22.1	22.3	18.8
ACCURACY	-0.7%	-8.0%	-12.7%	-17.0%
PRECISION (%RSD)	5.1%	6.3%	15.5%	7.7%
TRI-X-100				
TX-A1-5-1[b]	26.3	21.3	27.0	19.8
TX-A2-5-1	1.2	0.8	2.4	1.8
TX-B1-6-1	30.2	26.0	29.9	22.7
TX-B2-6-1	0.3	0.4	2.2	1.4
TX-G1-15-1	24.1	26.2	24.0	18.2
TX-G2-15-1	1.1	1.2	2.2	1.4
TX-H1-16-1	21.2	17.3	21.5	16.3
TX-H2-16-1	1.2	1.2	2.5	1.5
AVERAGE	26.4	23.6	27.9	20.8
ACCURACY	-7.2%	-1.8%	9.2%	-8.5%
PRECISION (%RSD)	13.1%	17.4%	12.6%	13.1%
Carboxen-564				
CB-A1-17-1	26.3	17.4	17.1	8.5
CB-A2-17-1	1.5	0.8	2.5	1.4
CB-B1-18-1	25.3	22.0	21.5	8.4
CB-B2-18-1	1.0	0.7	2.5	1.4
CB-C1-19-1	21.7	18.8	23.9	8.9
CB-C2-19-1	0.7	0.7	2.5	1.4
CB-D1-20-1	14.4	11.8	12.2	6.3
CB-D2-20-1	1.3	0.9	3.1	1.6
AVERAGE (w/o 20)[c]	25.5	20.2	23.4	10.0
ACCURACY	-10.4%	-16.1%	-8.6%	-55.9%
PRECISION (%RSD)	11.1%	11.4%	14.8%	2.8%

[a] Tedlar Bag Sample Code = Sample Collection Device - Orifice No. - Run No.
[b] Tube Sample Code = Sample Media - Quad Train/Tube No. (primary or secondary) - Orifice No. - Run No.
[c] Sample CB-D-20-1 was an outlier due to orifice malfunction.

TABLE 15. RUN 3 TEDLAR BAG SAMPLE RESULTS FOR SPIKED ORGANIC COMPOUNDS

	Chlorinated Organic Compound (ppm)			
	$CHCl_3$	CCl_4	TCE	PERC
Run 3 Calculated In-Stack Concentration	29.4	24.8	26.4	23.4
Small Tedlar Bags				
SB-1-3[a]	33.1	26.2	22.8	20.1
SB-2-3	31.4	19.9	18.7	16.8
SB-3-3	23.0	21.9	19.0	20.3
SB-4-3	21.3	15.8	16.6	13.9
AVERAGE SMALL BAG	27.2	21.0	19.3	17.8
ACCURACY	-7.4%	-15.4%	-26.9%	-23.9%
PRECISION (%RSD)	21.7%	20.8%	13.5%	17.3%
Large Tedlar Bags				
LB-7-3	31.8	22.7	26.1	22.3
LB-8-3	-0.3	-2.7	2.6	8.5
LB-9-3	41.8	36.2	34.3	31.4
LB-10-3	28.2	20.2	23.9	20.6
AVERAGE LARGE BAG	33.9	26.4	28.1	24.8
ACCURACY	15.6%	6.5%	6.6%	6.0%
PRECISION (%RSD)	20.8%	32.6%	19.3%	23.3%
TOTAL AVERAGE	30.1	23.3	23.1	20.8
ACCURACY	2.5%	-6.0%	-12.5%	-11.1%
PRECISION (%RSD)	22.8%	28.0%	25.8%	26.3%

[a] Tedlar Bag Sample Code = Sample Collection Device - Orifice No. - Run No.

TABLE 16. RUN 3 TUBE SAMPLE RESULTS FOR SPIKED ORGANIC COMPOUNDS

	Chlorinated Organic Compound (ppm)			
	$CHCl_3$	CCl_4	TCE	PERC
Run 3 Calculated In-Stack Concentration	29.4	24.8	26.4	23.4
TRI-X-100				
TX-5-A1-3[a]	30.2	22.9	27.2	23.1
TX-5-A2-3	*	0.0	0.0	0.0
TX-6-B1-3	63.8	23.7	32.7	28.3
TX-6-B2-3	*	0.0	0.0	0.0
TX-11-C1-3	47.5	2.1	15.8	8.0
TX-11-C2-3	*	17.1	10.8	15.0
TX-12-D1-3	45.6	2.4	16.1	6.7
TX-12-D2-3	*	18.1	11.6	16.0
AVERAGE	46.8	22.3	28.5	24.3
ACCURACY	59.4%	-9.8%	8.2%	3.9%
PRECISION (%RSD)	29.4%	14.5%	9.8%	11.1%
Carboxen-564				
CB-13-A1-3	30.1	22.7	24.2	22.7
CB-13-A2-3	0.0	0.0	0.0	-0.9
CB-14-B1-3	27.2	29.2	25.7	23.2
CB-14-B2-3	0.0	0.0	0.0	-0.2
CB-15-C1-3	25.3	25.9	23.8	22.5
CB-15-C2-3	0.0	0.0	0.0	-0.9
CB-16-D1-3	32.2	28.8	20.1	21.1
CB-16-D2-3	0.0	0.0	0.0	-0.9
CB-17-E1-3	20.1	26.8	22.1	13.9
CB-17-E2-3	0.0	0.0	0.0	0.0
CB-18-F1-3	23.5	28.4	22.7	14.0
CB-18-F2-3	0.0	0.0	0.0	0.0
AVERAGE	26.4	27.0	23.1	22.4**
ACCURACY	-10.0%	8.9%	-12.4%	-4.3%
PRECISION (%RSD)	16.7%	9.0%	8.3%	4.0%

[a]Tube Sample Code = Sample Media - Quad Train/Tube No. (primary or secondary) - Orifice No. - Run No.

*Peak interference in analysis (shoulder).

**Does not include CB-17-1-3 and CB-18-F1-3.

TABLE 17. RUN 4 TUBE SAMPLE RESULTS FOR SPIKED ORGANIC COMPOUNDS

	Chlorinated Organic Compound (ppm)			
	$CHCl_3$	CCl_4	TCE	PERC
Run 4 Calculated In-stack Concentration	59.0	49.8	53.0	47.0
Carboxen-564[a]				
CB-13-A1-4	33.6	44.0	35.0	19.4
TX-13-A2-4	*	0.0	0.0	0.0
CB-14-B1-4	42.7	46.5	50.1	37.0
TX-14-B2-4	*	0.0	0.0	0.0
CB-15-C1-4	77.9	60.7	43.8	37.0
TX-15-C2-4	*	0.0	0.0	0.0
CB-16-D1-4	46.0	55.3	46.4	17.1
TX-16-D2-4	*	0.0	0.0	0.0
AVERAGE	50.1	51.6	43.8	27.6
ACCURACY	-15.2%	3.7%	-17.3%	-41.2%
PRECISION (%RSD)	38.6%	15.0%	14.7%	39.3%
TRI-X-100				
TX-5-A1-4	35.2	12.2	51.3	24.4
TX-5-A2-4	0.0	9.0	1.8	2.6
TX-6-B1-4	39.3	10.7	42.2	26.5
TX-6-B2-4	0.0	12.9	8.6	8.1
TX-11-C1-4	38.6	7.2	29.0	12.1
TX-11-C2-4	0.0	17.4	22.1	25.7
TX-12-D1-4	39.1	11.6	30.8	13.8
TX-12-D2-4	0.0	11.5	15.1	12.8
TX-19-E1-4	29.7	12.8	35.9	22.8
TX-19-E2-4	20.3	0.0	16.2	22.2
TX-20-F1-4	37.3	8.2	14.1	12.4
TX-20-F2-4	0.0	22.9	36.7	33.7
AVERAGE	39.9	22.7	50.6	37.8
ACCURACY	-32.4%	-54.4%	-4.5%	-19.5%
PRECISION (%RSD)	11.6%	25.8%	4.3%	19.0%

[a]Back up tubes were TRI-X-100.
[b]Tube Sample Code = Sample Media - Quad Train/Tube No. (primary or secondary) - Orifice No. - Run No.
*Second tube analysis resulted in questionable data.

TABLE 18. RUN 5 TUBE RESULTS FOR SPIKED ORGANIC COMPOUNDS

	Chlorinated Organic Compound (ppm)			
	$CHCl_3$	CCl_4	TCE	PERC
Run 5 Calculated In-Stack Concentration	29.5	24.9	26.5	23.5
TRI-X-100				
TX-5-A1-5[a]	53.6	8.9	23.2	16.3
TX-5-A2-5	*	2.7	0.0	1.8
TX-6-B1-5	49.0	10.6	21.4	15.5
TX-6-B2-5	*	8.3	0.0	0.0
TX-11-C1-5	22.7	16.7	20.7	16.3
TX-11-C2-5	*	8.8	5.7	6.0
TX-12-D1-5	27.9	21.5	25.4	21.5
TX-12-D2-5	*	3.4	0.6	0.2
TX-19-E1-5	29.1	21.1	27.0	21.6
TX-19-E2-5	1.8	1.5	0.0	0.0
TX-20-F1-5	29.2	20.6	27.2	22.9
TX-20-F2-5	5.1	5.4	0.8	0.0
AVERAGE	29.0	21.6	25.4	20.3
ACCURACY	-1.8%	-13.4%	-4.3%	-13.4%
PRECISION (%RSD)	17.0%	25.8%	9.9%	14.2%
Carboxen-564				
CB-13-A1-5	28.3	26.3	19.6	17.3
CB-13-A2-5	2.1	7.7	4.0	0.0
CB-14-B1-5	24.8	23.8	19.9	17.5
CB-14-B2-5	0.0	0.0	0.0	0.0
CB-15-C1-5	31.0	24.9	22.8	19.1
CB-15-C2-5	3.9	10.4	0.0	0.0
CB-16-D1-5	28.8	22.3	17.1	10.3
CB-16-D2-5	0.0	0.0	0.0	0.0
AVERAGE	29.7	28.9	20.8	16.0
ACCURACY	0.7%	15.9%	-21.5%	-31.8%
PRECISION (%RSD)	14.1%	23.4%	14.3%	24.4%

[a]Tube Sample Code = Sample Media - Quad Train/Tube No. (primary or secondary) - Orifice No. - Run No.

SECTION 7
QUALITY ASSURANCE/QUALITY CONTROL

This section discusses the quality assurance and quality control (QA/QC) procedures implemented for the field test evaluations of Method 18 as applied to chlorinated solvent emissions testing. QA/QC procedures are necessary to document and quantify the acceptability and reliability of the data generated. The QA/QC guidelines emphasized the following areas: data quality objectives, adherence to prescribed sampling and analytical techniques, data recording, sample custody, and calibration procedures. The QA/QC activities and results are described in the following subsections.

SAMPLING QA/QC ACTIVITIES

Quality control for gas sampling for both field evaluation tests emphasized: (1) equipment checks, (2) procedural quality control checks, and (3) sample custody procedures.

Pretest equipment checks were conducted according to Method 18 sampling procedure quality assurance. Pre- and posttest calibrations were conducted on all flow meters and orifices used in the sampling or spiking systems. All equipment met the calibration criteria specified in Method 18. All sample lines were purged for approximately 5 minutes prior to the injection of the solvents into the stack. The stack velocity was monitored during the entire 60 minute period for each sampling run.

Each portion of the sampling train was visually inspected for proper assembly before the sampling runs began. All sampling data and calculations were recorded on preformatted data sheets, with any unusual occurrence being noted during each run on the appropriate data form. During both field evaluation tests, all sampling trains met quality assurance objectives and were leak free prior to sampling.

Sample custody procedures for the field evaluations were based on generally recommended procedures. Since samples were analyzed in the

laboratory upon completion of the sampling run, emphasis was placed on careful labeling of samples collected. All samples were given a unique alphanumeric sample identification code. Sample labels were prepared prior to field evaluation tests and upon sample completion were affixed to each sample container or tube. Samples were immediately transported from the sampling location to the laboratory for analysis.

ANALYTICAL QA/QC ACTIVITIES

All analyses for both test programs were performed using accepted laboratory procedures in accordance with specified analytical protocols. The specified quality control procedures for sample preparatory work and sample analysis by GC/FID are discussed below.

Standards for Tedlar bag samples were prepared as outlined in Section 4. Calibration data were collected for the ranges of the expected field samples. Standards for desorbed solutions were prepared using 0.5 - 36 ul of solvent in 4 ml of methylene chloride. From these standards, working calibration curves were calculated as described in Method 18. Immediately prior to analysis of the stack gas samples, audit analyses were performed for each solvent. The audit analyses agreed within 10% for all components of interest. The analysis of cylinder audit gases provided by QAD also allowed estimation of accuracy. For the first field evaluation test, the audit performed in the 10-100 ppm range differed from its certified value by 6.0% and the audit performed in the range of 100-1000 ppm differed from its certified value by 7.0%. During the second field evaluation test, an audit for each solvent was performed. These audit results are reported in Table 19.

TABLE 19. AUDIT VALUES FOR SECOND FIELD EVALUATION TEST

Solvent	Entropy Value (ppm)	Actual Value (ppm)	% Accuracy
Carbon tetrachloride	7.28	8.03	- 9.3
Chloroform	3.27	4.12	-20.6*
Perchloroethylene	7.30	8.03	-10.9
Trichloroethylene	9.43	10.20	7.54

*The chloroform audit cylinder concentration was below the lowest concentration included in the calibration curve.

REFERENCES

1. "Method 18: Measurement of Gaseous Organic Compound Emissions by Gas Chromatography," 40 Code of Federal Regulations 60, July 1, 1987.

2. Knoll, Joseph E., Mark A. Smith, and M. Rodney Midgett, "Evaluation of Emission Test Methods for Halogenated Hydrocarbons - Vol. 1, CCl_4 $C_2H_4Cl_2$, C_2Cl_4, and C_2HCl_3," EPA-600/4-79-02, U. S. Environmental Protection Agency, Research Triangle Park, North Carolina.

3. Quality Assurance Handbook for Air Pollution Measurement Systems, Vol. III, EPA-600/4-77-027b, August 1977; Section 3.16, "Method 18: Measurement of Gaseous Organic Compound Emissions by Gas Chromatography."

4. Knoll, Joseph E., Journal of Chromatographic Science, 1985, Vol. 23, 422-425.

5. Bradd, Byron B., "Field Validation of EPA Reference Method 23," report prepared for U. S. Environmental Protection Agency under Contract No. 68-02-3405.

6. Goodrich, J. D., W. G. DeWees, and R. R. Segall, "Sampling and Analysis of Butadiene at a Synthetic Rubber Plant," Report prepared for U. S. Environmental Protection Agency under Contract No. 68-02-4442.

APPENDIX A

ANALYTICAL DATA SHEETS

Perchloroethylene Method Analytical Data Sheet

Job Name: PERC — Job Num. 4120
Analyst: JD Goodrich — Date: Dec. 10, 1987

Perchloroethylene Standard Calibration Curve by Linear Regression

Charge (ul)	Peak Area Run 1	Peak Area Run 2	Peak Area Average	Predicted Charge (ul)	Percent Deviation from Actual
50.0	2189	2207	2198	52.0	4.1%
100.0	4634	4452	4543	96.9	-3.1%
200.0	9849	10107	9978	201.0	0.5%

Standard Curve Slope 52.22 Intercept -519.26

Desorption Coefficient: 0.82 +/- 0.04

Sample ID	Run 1 Area	Run 2 Area	Average	Percent Deviation	Detected Charge (ul)	Flow Rate (L/min)	PERC Conc. (ppm)
Run time: 60 minutes							
AO-5	6793	7079	6936	2.1%	143	1.32	517
AO-6	7288	7585	7437	2.0%	152	1.30	561
AO-7	6866	6727	6796	1.0%	140	1.34	500
AO-8	6918	7249	7084	2.3%	146	1.33	524
Average +/- 95% Confidence Interval					526 +/-	41	
AO-5-B	23						
AO-8-B	ND						
Run time: 60 minutes							
CO-5	1964	1902	1933	1.6%	47	1.32	170
CO-6	1658	1693	1675	1.0%	42	1.30	155
CO-7	1878	1770	1824	2.9%	45	1.34	160
CO-8	1906	1989	1947	2.1%	47	1.33	170
Average +/- 95% Confidence Interval					164 +/-	12	
Run time: 60 minutes							
BI-8	9877	10093	9985	1.1%	201	0.0794	12120
Run time: 60 minutes							
DI-8	8714	8965	8840	1.4%	179	0.0794	10799

Perchloroethylene Method Analytical Data Sheet

Job Name: PERC — Job Num. 4120
Analyst: JD Goodrich — Date: Dec. 11, 1987

Perchloroethylene Standard Calibration Curve by Linear Regression

Charge (ul)	Peak Area Run 1	Peak Area Run 2	Peak Area Average	Predicted Charge (ul)	Percent Deviation from Actual
50.0	2265	2349	2307	49.85	-0.29%
100.0	4839	5229	5034	100.24	0.24%
180.0	9470	9223	9346	179.91	-0.05%

Standard Curve Slope 54.12 Intercept -390.87

Desorption Coefficient: 0.82 +/- 0.04

Sample ID	Run 1 Area	Run 2 Area	Average	Percent Deviation	Detected Charge (ul)	Flow Rate (L/min)	PERC Conc. (ppm)
Run time: 60 minutes							
GO-5	2182	2275	2229	2.09%	48	1.32	175
GO-6	2193	2084	2139	2.54%	47	1.30	172
GO-7	2273	2297	2285	0.52%	49	1.34	177
GO-8	2183	2127	2155	1.32%	47	1.33	169
Average +/- 95% Confidence Interval					173 +/-	6	
Run time: 60 minutes							
JO-5	7325	7736	7531	2.73%	146	1.32	530
JO-6	7436	7776	7606	2.24%	148	1.30	544
JO-7	8333	8054	8194	1.71%	159	1.34	566
JO-8	7336	7754	7545	2.76%	147	1.33	527
Average +/- 95% Confidence Interval					542 +/-	33	
Run time: 60 minutes							
**** Note: Injection volume was increased from 2.00 ul to 5.00 ul ***							
FI-8	5185	5234	5210	0.48%	41	0.0794	2721
Run time: 60 minutes							
HI-8	7550	7304	7427	1.65%	144	0.0794	8703
Run time: 60 minutes							
KI-8	9417	9725	9571	1.61%	184	0.0794	11090

Perchloroethylene Method Analytical Data Sheet

Job Name: PERC — Job Num. 4120
Analyst: JD Goodrich — Date: Dec. 12, 1987

Perchloroethylene Standard Calibration Curve by Linear Regression

Charge (ul)	Peak Area Run 1	Peak Area Run 2	Peak Area Average	Predicted Charge (ul)	Percent Deviation from Actual
50.0	2273	2458	2366	50.6	1.1%
100.0	4811	4768	4789	99.1	-0.9%
180.0	8516	9181	8849	180.4	0.2%

Standard Curve Slope 49.95 Intercept -159.66

Description Coefficient: 0.82 +/- 0.04

Sample ID	Run 1 Area	Run 2 Area	Average	Percent Deviation	Detected Charge (ul)	Flow Rate (L/min)	PERC Conc. (ppm)
Run time: 55 minutes							
LC-5	3187	3313	3250	1.9%	68	1.32	270
LC-6	3059	3088	3073	0.5%	65	1.30	260
LC-7	2825	2865	2845	0.7%	60	1.34	234
LC-8	2971	2871	2921	1.7%	62	1.33	242
Average +/- 95% Confidence Interval					252 +/-	26	
Run time: 60 minutes							
NC-5	3278	3321	3299	0.7%	69	1.32	251
NC-6	3036	3026	3031	0.2%	64	1.30	235
NC-7	3228	3209	3218	0.3%	68	1.34	241
NC-8	3037	3222	3129	3.0%	66	1.33	237
Average +/- 95% Confidence Interval					241 +/-	11	
Run time: 60 minutes							
**** Note: Injection volume was decreased from 2.00 ul to 1.00 ul ***							
$QC-5	5080	5048	5064	0.3%	209	1.32	758
$QC-6	4885	5204	5045	3.2%	208	1.30	767
$ Capacity of primary portion exceeded							
%QC-7	4444	4699	4572	2.8%	189	6.12	148
%QC-8	4392	4279	4335	1.3%	180	6.03	143
% Capacity exceeded; breakthrough likely							

Perchloroethylene Method Analytical Data Sheet

Job Name: PERC
Analyst: JD Goodrich

Job Num. 4120
Date: Dec. 13, 1987

Perchloroethylene Standard Calibration Curve by Linear Regression

Charge (ul)	Peak Area Run 1	Run 2	Average	Predicted Charge (ul)	Percent Deviation from Actual
50.0	2356	2308	2332	49.65	-0.71%
100.0	5170	5034	5102	100.57	0.57%
180.0	9441	9378	9410	179.78	-0.12%

Standard Curve Slope 54.39 Intercept -368.10

Desorption Coefficient: 0.82 +/- 0.04

Sample ID	Run 1 Area	Run 2 Area	Average	Percent Deviation	Detected Charge (ul)	Flow Rate (L/min)	PERC Conc. (ppm)
Run time: 55 minutes							
MI-8	4023	4079	4051	0.69%	81	0.0794	5341
Run time: 60 minutes							
PI-8	6024	6221	6122	1.61%	119	0.0794	7190
Run time: 60 minutes							
RI-8	7424	7692	7558	1.77%	146	0.0794	8781
BACKUPS							
JO-7-B	ND						
JO-8-B	ND						
KI-7-B	ND						
KI-8-B	ND						
QO-5-B	2318	2362	2340	0.94%	50	1.32	180
QO-6-B	1981	1818	1899	4.29%	42	1.30	153
QO-7-B	2830	2752	2791	1.39%	58	6.03	46
QO-8-B	2757	2738	2747	0.34%	57	6.03	45

Chloronated Solvents Method Analytical Data Sheet

Job Name: Solvents CCl4 — Job Num. 4154
Analyst: AC Carver — Date: Jan 2, 1989

Carbon tetrachloride Standard Calibration Curve by Linear Regress

CCl4 Charge (ul)	Peak Area Run 1	Run 2	Average	Predicted Charge (ul)	Percent Deviation from Actual
0.50	2896		2896	0.55	10.13%
1.00	5542		5542	0.90	-10.13%
1.50	10497		10497	1.55	3.38%

Standard Curve Slope 7601.00 Intercept -1289

Desorption Coefficient: 0.82 +/- 0.04

Sample ID	Run 1 Area	Run 2 Area	Average	Percent Deviation	Detected Charge (ul)	Flow Rate (L/min)	CCl4 Conc. (ppm)
Run time (MIN.)		60					
CB-13-A1-3	7954	7894	7924	0.38%	1.21	0.240	25.6
CB-14-B1-3	112493		112493	100.00%	14.97	0.240	316.1
TX-B1-12-2	25620	24819	25220	1.59%	3.49	0.238	74.3
TX-B2-12-2	54964	50861	52913	3.88%	7.13	0.238	151.8
TX-C1-13-2	20640	17158	18899	9.21%	2.66	0.233	57.8
TX-C2-13-2	84507	74944	79726	6.00%	10.66	0.233	231.8
TX-D1-14-2	7333		7333	100.00%	1.13	0.240	24.0
TX-D2-14-2	70394		70394	100.00%	9.43	0.239	200.0
	EXPECTED	81.5	AVERAGE	270.3 +/- 9.2%		231.8%	
CB-B1-16-2	127543		127543	100.00%	16.95	0.239	286.8
CB-B2-16-2	68653		68653	100.00%	9.20	0.239	155.7
CB-C1-17-2	167983		167983	100.00%	22.27	0.240	375.3
CB-C2-17-2	18715		18715	100.00%	2.63	0.240	44.3
CB-D1-18-2	147093	152627	149860	1.85%	19.89	0.232	346.7
CB-D2-18-2	0		0	ERR	0.17	0.232	3.0
	EXPECTED	81.5				429.1%	

Chloronated Solvents Method Analytical Data Sheet

Job Name: Solvents CC14 — Job Num. 4154
Analyst: AC Carver — Date: Feb. 5, 1989

Carbon tetrachloride Standard Calibration Curve by Linear Regressio

CC14 Charge (ppm)	Peak Area Run 1	Run 2	Average	Predicted Charge (ppm)	Percent Deviation from Actual
9.97	572	555	564	10.26	2.91%
17.53	811	959	885	17.17	-2.04%
50.10	2430	2409	2420	50.17	0.13%

Standard Curve Slope 46.51 Intercept 86

Desorption Coefficient: 0.82 +/- 0.04

Sample ID	Run 1 Area	Run 2 Area	Average	Percent Deviation	Detected Charge (ul)	Flow Rate (L/min)	CC14 Conc. (ppm)
Run time (MIN.)		40					
SB-2-5	1339	1339	1339	0.00%	26.93	0.242	26.9
SB-3-5	306	283	295	3.90%	4.48	0.244	4.5
SB-4-5	1251	1207	1229	1.79%	24.57	0.242	24.6
audit #AAL	437	413	425	2.82%	7.28	0.240	195.0

Chloronated Solvents Method Analytical Data Sheet

Job Name: Solvents CC14 Job Num. 4154
Analyst: AC Carver Date: Feb 6, 1989

Carbon tetrachloride Standard Calibration Curve by Linear Regress

CC14 Charge (ul)	Peak Area Run 1	Run 2	Average	Predicted Charge (ul)	Percent Deviation from Actual
0.50	22243		22243	0.49	-2.19%
1.00	47148		47148	1.02	2.19%
1.50	68979		68979	1.49	-0.73%

Standard Curve Slope 46736.00 Intercept -613

Desorption Coefficient: 0.82 +/- 0.04

Sample ID	Run 1 Area	Run 2 Area	Average	Percent Deviation	Detected Charge (ul)	Flow Rate (L/min)	CC14 Conc. (ppm)
Run time (MIN.)		60					
TX-5-A1-3	50663	46169	48416	4.64%	1.05	0.232	22.9
TX-5-A2-3	0	0	0	ERR	0.01	0.232	
TX-6-B1-3	51254	49892	50573	1.35%	1.10	0.234	23.7
TX-6-B2-3	5691		5691	100.00%	0.13	0.234	2.9
TX-11-C1-3	4145	3878	4012	3.33%	0.10	0.235	2.1
TX-11-C2-3	41050	32062	36556	12.29%	0.80	0.235	17.1
TX-12-D1-3	4725		4725	100.00%	0.11	0.238	2.4
TX-12-D2-3	40505	37879	39192	3.35%	0.85	0.238	18.1
EXPECTED		24.8	AVERAGE	22.3 +/-	14.5%	-9.8%	
CB-13-A1-3	67748	53304	60526	11.93%	1.31	0.233	22.7
CB-13-A2-3	0		0	ERR	0.01	0.233	
CB-14-B1-3	80278		80278	100.00%	1.73	0.240	29.2
CB-14-B2-3	0	0	0	ERR	0.01	0.240	
CB-15-C1-3	70708		70708	100.00%	1.53	0.238	25.9
CB-15-C2-3	0		0	ERR	0.01	0.238	
CB-16-D1-3	78850		78850	100.00%	1.70	0.239	28.8
CB-16-D2-3	0		0	ERR	0.01	0.239	
CB-17-E1-3	73453		73453	100.00%	1.58	0.239	26.8
CB-17-E2-3	0						
CB-18-F1-3	77874		77874	100.00%	1.68	0.239	28.4
CB-18-F2-3	0						
EXPECTED		24.8	AVERAGE	27.0 +/-	9.0%	8.9%	

Chloronated Solvents Method Analytical Data Sheet

Job Name: Solvents CC14 — Job Num. 4154
Analyst: AC Carver — Date: Feb 6, 1989

Carbon tetrachloride Standard Calibration Curve by Linear Regress

CC14 Charge (ul)	Peak Area Run 1	Peak Area Run 2	Peak Area Average	Predicted Charge (ul)	Percent Deviation from Actual
0.50	22243		22243	0.49	-2.19%
1.00	47148		47148	1.02	2.19%
1.50	68979		68979	1.49	-0.73%

Standard Curve Slope 46736.00 Intercept -613

Desorption Coefficient: 0.82 +/- 0.04

Sample ID	Run 1 Area	Run 2 Area	Average	Percent Deviation	Detected Charge (ul)	Flow Rate (L/min)	CC14 Conc. (ppm)
Run time (MIN.)		60					
TX-5-A1-5	19026	17756	18391	3.45%	0.41	0.232	8.9
TX-5-A2-5	5428	4799	5114	6.15%	0.12	0.232	2.7
TX-6-B1-5	24346	20010	22178	9.78%	0.49	0.234	10.6
TX-6-B2-5	17799	16968	17384	2.39%	0.39	0.234	8.3
TX-11-C1-5	35537	35585	35561	0.07%	0.77	0.235	16.7
TX-11-C2-5	18632	18402	18517	0.62%	0.41	0.235	8.8
TX-12-D1-5	48591	44449	46520	4.45%	1.01	0.238	21.5
TX-12-D2-5	6949		6949	100.00%	0.16	0.238	3.4
TX-19-E1-5	48276	42943	45610	5.85%	0.99	0.238	21.1
TX-19-E2-5	2630		2630	100.00%	0.07	0.238	1.5
TX-20-F1-5	45396	43742	44569	1.86%	0.97	0.238	20.6
TX-20-F2-5	11307		11307	100.00%	0.26	0.238	5.4
EXPECTED		24.9	AVERAGE	21.6 +/-	25.8%	-13.4%	
CB-13-A1-5	72690	67776	70233	3.50%	1.52	0.233	26.3
CB-13-A2-5	20069		20069	100.00%	0.44	0.233	7.7
CB-14-B1-5	66380	64460	65420	1.47%	1.41	0.240	23.8
CB-14-B2-5	0	0	0		0.01	0.240	
CB-15-C1-5	69404	66586	67995	2.07%	1.47	0.238	24.9
CB-15-C2-5	27978		27978	100.00%	0.61	0.238	10.4
CB-16-D1-5	63851	58292	61072	4.55%	1.32	0.239	22.3
CB-16-D2-5	0		0		0.01	0.239	
EXPECTED		24.9	AVERAGE	28.9 +/-	23.4%	15.9%	

Chloronated Solvents Method Analytical Data Sheet

Job Name: Solvents CHCl3 Job Num. 4154
Analyst: AC Carver Date: Dec. 30, 1988

Chloroform Standard Calibration Curve by Linear Regression

Charge (ul)	Peak Area Run 1	Run 2	Average	Predicted Charge (ul)	Perc Devia from A
0.50	31073		31073	0.48	-3.33%
1.00	66640		66640	1.03	3.33%
1.50	95753		95753	1.48	-1.11%

Standard Curve Slope 64680 Intercept -191

Desorption Coefficient: 1.0 +/- 0.04

Sample ID	Run 1 Area	Run 2 Area	Average	Percent Deviation	Detected Charge (ul)	Flow Rate (L/min)	CHCl3 Conc. (ppm)
Run time (min):		60					
TX-5-A1-5	164217	155004	159611	2.89%	2.47	0.232	53.6
TX-5-A2-5	*	*	0	ERR	0.00	0.232	*
TX-6-B1-5	153955	139917	146936	4.78%	2.27	0.234	49.0
TX-6-B2-5	*	*	0	ERR	0.00	0.234	*
TX-11-C1-5	67667	69130	68399	1.07%	1.06	0.235	22.7
TX-11-C2-5	*	*	0	ERR	0.00	0.235	*
TX-12-D1-5	87058	83222	85140	2.25%	1.32	0.238	27.9
TX-12-D2-5	*		0	ERR	0.00	0.238	*
TX-19-E1-5	89535	81446	85491	4.73%	1.32	0.229	29.1
TX-19-E2-5	5128	5128	5128	0.00%	0.08	0.229	1.8
TX-20-F1-5	87606	84391	85999	1.87%	1.33	0.23	29.2
TX-20-F2-5	14926		14926	100.00%	0.23	0.23	5.1
EXPECTED		29.5	AVERAGE	29.0 +/-	17.0%	-1.8%	
******DOES NOT INCLUDE TX-5-A1-5,TX-5-A2-5,TX-6-B1-5******							
CB-13-A1-5	85458	83476	84467	1.17%	1.31	0.233	28.3
CB-13-A2-5	6072		6072	100.00%	0.10	0.233	2.1
CB-14-B1-5	76579	75831	76205	0.49%	1.18	0.24	24.8
CB-14-B2-5	0	0	0		0.00	0.24	
CB-15-C1-5	96244	92817	94531	1.81%	1.46	0.238	31.0
CB-15-C2-5	11733		11733	100.00%	0.18	0.238	3.9
CB-16-D1-5	92361	83957	88159	4.77%	1.37	0.239	28.8
CB-16-D2-5	0		0		0.00	0.239	
EXPECTED		29.5	AVERAGE	29.7 +/-	14.1%	0.7%	

Chloronated Solvents Method Analytical Data Sheet

Job Name:Solvents CHCl3 Job Num. 4154
Analyst: AC Carver Date: Dec. 30, 1988

Chloroform Standard Calibration Curve by Linear Regression

Charge (ul)	Peak Area Run 1	Run 2	Average	Predicted Charge (ul)	Perc Devia from A
0.50	19857		19857	0.57	13.61%
1.00	39669		39669	0.86	******
1.50	86816		86816	1.57	4.54%

Standard Curve Slope 66959 Intercept -18178

Desorption Coefficient: 1.0 +/- 0.04

Sample ID	Run 1 Area	Run 2 Area	Average	Percent Deviation	Detected Charge (ul)	Flow Rate (L/min)	CHCl3 Conc. (ppm)
Run time (min):		60					
CB-13-A1-4	91963	79898	85931	7.02%	1.55	0.233	33.6
CB-14-B1-4	125429	110679	118054	6.25%	2.03	0.240	42.7
TX-14-B2-4	***	73945	36973	100.00%	0.82	0.240	17.3
CB-15-C1-4	226598	230211	228405	0.79%	3.68	0.238	77.9
TX-15-C2-4	***	97245	48623	100.00%	1.00	0.238	21.1
CB-16-D1-4	134635	121260	127948	5.23%	2.18	0.239	46.0

EXPECTED 59.0 AVERAGE 50.1 +/- 38.6% -15.2%

Chloronated Solvents Method Analytical Data Sheet

Job Name: Solvents CHC13 Job Num. 4154
Analyst: AC Carver Date: Jan 2, 1989

Chloroform Standard Calibration Curve by Linear Regression

Charge (ul)	Peak Area Run 1	Run 2	Average	Predicted Charge (ul)	Perc Devia from A
0.50	19857		19857	0.57	13.61%
1.00	39669		39669	0.86	******
1.50	86816		86816	1.57	4.54%

Standard Curve Slope 66959 Intercept -18178

Desorption Coefficient: 1.0 +/- 0.04

Sample ID	Run 1 Area	Run 2 Area	Average	Percent Deviation	Detected Charge (ul)	Flow Rate (L/min)	CHC13 Conc. (ppm)
Run time (min):		60					
CB-13-A1-3	71938	70532	71235	0.99%	1.34	0.233	28.9
TX-13-A2-3	****		0	ERR	0.27	0.233	5.9
CB-14-B1-3	64651	64651	64651	0.00%	1.24	0.240	26.0
TX-14-B2-3	***	73945	36973	100.00%	0.82	0.240	17.3
CB-15-C1-3	58017	58017	58017	0.00%	1.14	0.238	24.1
TX-15-C2-3	***	97245	48623	100.00%	1.00	0.238	21.1
CB-16-D1-3	76089	84216	80153	5.07%	1.47	0.239	30.9
TX-16-D2-3	***		0	ERR	0.27	0.239	5.7
CB-17-E1-3	91044	43827	67436	35.01%	1.28	0.240	26.8
TX-17-E2-3	***	97245	48623	100.00%	1.00	0.240	20.9
CB-18-F1-3	94051	60481	77266	21.72%	1.43	0.232	30.9
TX-18-F2-3	***		0	ERR	0.27	0.239	5.7

EXPECTED 29.4 AVERAGE 27.9 +/- 10.5% -4.8%

Chloronated Solvents Method Analytical Data Sheet

Job Name: Solvents CHC13 — Job Num. 4154
Analyst: AC Carver — Date: Jan. 2, 1989

Chloroform Standard Calibration Curve by Linear Regression

Charge (ul)	Peak Area Run 1	Run 2	Average	Predicted Charge (ul)	Perc Devia from A
0.50	19857		19857	0.57	13.61%
1.00	39669		39669	0.86	******
1.50	86816		86816	1.57	4.54%

Standard Curve Slope 66959 Intercept -18178

Desorption Coefficient: 1.0 +/- 0.04

Sample ID	Run 1 Area	Run 2 Area	Average	Percent Deviation	Detected Charge (ul)	Flow Rate (L/min)	CHC13 Conc. (ppm)
Run time (min):		60					
CB-13-A1-4	91963	79898	85931	7.02%	1.55	0.233	33.6
TX-13-A2-4	****		0	ERR	0.27	0.233	5.9
CB-14-B1-4	125429	110679	118054	6.25%	2.03	0.240	42.7
TX-14-B2-4	***	73945	36973	100.00%	0.82	0.240	17.3
CB-15-C1-4	226598	230211	228405	0.79%	3.68	0.238	77.9
TX-15-C2-3	***	97245	48623	100.00%	1.00	0.238	21.1
CB-16-D1-3	134635	121260	127948	5.23%	2.18	0.239	46.0
TX-16-D2-3	***		0	ERR	0.27	0.239	5.7
CB-17-E1-3	94999	43827	69413	36.86%	1.31	0.240	27.5
TX-17-E2-3	***	97245	48623	100.00%	1.00	0.240	20.9
CB-18-F1-3	98006	64436	81221	20.67%	1.48	0.232	32.2
TX-18-F2-3	***		0	ERR	0.27	0.239	5.7
EXPECTED		59.0	AVERAGE	50.1 +/- 38.6%		-15.2%	

Chloronated Solvents Method Analytical Data Sheet

Job Name: Solvents CHCl3 Job Num. 4154
Analyst: AC Carver Date: Feb 2, 1989

Chloroform Standard Calibration Curve by Linear Regression

Charge (ul)	Peak Area Run 1	Run 2	Average	Predicted Charge (ul)	Perc Devia from A
0.50	19857		19857	0.57	13.61%
1.00	39669		39669	0.86	******
1.50	86816		86816	1.57	4.54%

Standard Curve Slope 66959 Intercept -18178

Desorption Coefficient: 1.0 +/- 0.04

Sample ID	Run 1 Area	Run 2 Area	Average	Percent Deviation	Detected Charge (ul)	Flow Rate (L/min)	CHCl3 Conc. (ppm)
Run time (min):		60					
CB-13-A1-3	75893	74487	75190	0.93%	1.39	0.233	30.1
CB-13-A2-3	0		0	ERR	0.27	0.233	
CB-14-B1-3	68600		68600	100.00%	1.30	0.24	27.2
CB-14-B2-3	0	0	0	ERR	0.27	0.24	
CB-15-C1-3	61972		61972	100.00%	1.20	0.238	25.3
CB-15-C2-3	0		0	ERR	0.27	0.238	
CB-16-D1-3	80044	88171	84108	4.83%	1.53	0.239	32.2
CB-16-D2-3	0		0	ERR	0.27	0.239	
CB-17-E1-3	46934	43827	45381	3.42%	0.95	0.238	20.1
CB-17-E2-3	0						
CB-18-F1-3	48006	64436	56221	14.61%	1.11	0.238	23.5
CB-18-F2-3	0						
EXPECTED		29.4	AVERAGE	26.4 +/-	16.7%	-10.0%	

Chloronated Solvents Method Analytical Data Sheet

Job Name: Solvents CHCl3 Job Num. 4154
Analyst: AC Carver Date: Feb. 5, 1989

Chloroform Standard Calibration Curve by Linear Regression

Charge (ppm)	Peak Area Run 1	Peak Area Run 2	Peak Area Average	Predicted Charge (ppm)	Percent Deviation from Actual
9.97	3572	3621	3597	10.83	8.61%
17.53	5014	5264	5139	16.47	-6.04%
50.00	14019	14697	14358	50.20	0.40%

Standard Curve Slope 273.33 Intercept 636.74

Desorption Coefficient: 1.0 +/- 0.04

Sample ID	Run 1 Area	Run 2 Area	Average	Percent Deviation	Detected Charge (PPM)	Flow Rate (L/min)	CHCl3 Conc. (ppm)
Run time (min):		40					
SB-1-5	425	426	426	0.12%	-0.77	0.242	-0.8
SB-2-5	10490	12036	11263	6.86%	38.88	0.242	38.9
SB-3-5	2952	2713	2833	4.22%	8.03	0.244	8.0
SB-4-5	9544	11551	10548	9.51%	36.26	0.246	36.3

EXPECTED 29.4 AVERAGE 11.8 +/- 162.5% -59.9%

AVERAGE SMALL BAGS 20.6 +/- 96.8% -29.8%

******DOES NOT INCLUDE LB-8-3******

Chloronated Solvents Method Analytical Data Sheet

Job Name: Solvents TCE Job Num. 4154
Analyst: AC Carver Date: Jan 2, 1989

Carbon tetrachloride Standard Calibration Curve by Linear Regress

TCE Charge (ul)	Peak Area Run 1	Run 2	Average	Predicted Charge (ul)	Percent Deviation from Actual
0.50	56001		56001	0.48	-3.04%
1.00	113359		113359	1.03	3.04%
1.50	161141		161141	1.48	-1.01%

Standard Curve Slope 105140 Intercept 5027

Desorption Coefficient: 0.82 +/- 0.04

Sample ID	Run 1 Area	Run 2 Area	Average	Percent Deviation	Detected Charge (ul)	Flow Rate (L/min)	TCE Conc. (ppm)
Run time(min):		60					
CB-13-A1-3	149738	115609	132674	12.86%	1.21	0.233	24.7
TX-13-A2-3	******		0	ERR	-0.05	0.233	-1.0
CB-14-B1-3	142136	107114	124625	14.05%	1.14	0.240	22.5
TX-14-B2-3	***	132259	66130	100.00%	0.58	0.240	11.5
CB-15-C1-3	132987	130036	131512	1.12%	1.20	0.238	24.0
TX-15-C2-3	***	177312	88656	100.00%	0.80	0.238	15.9
CB-16-D1-3	117236	135292	126264	7.15%	1.15	0.239	22.9
TX-16-D2-3	***		0	ERR	-0.05	0.239	-0.9
CB-17-E1-3	90520	116522	103521	12.56%	0.94	0.240	18.5
TX-17-E2-3	***	177312	88656	100.00%	0.80	0.240	15.7
CB-18-F1-3	122026	122026	122026	0.00%	1.11	0.232	22.8
TX-18-F2-3	***		0	ERR	-0.05	0.239	-0.9
	EXPECTED	26.4	AVERAGE	29.7 +/- 25.6%		12.8%	
CB-A2-15-2	3566	5382	4474	20.30%	-0.01	0.242	-0.1
CB-B1-16-2	238228		238228	100.00%	2.22	0.239	42.7
CB-B2-16-2	0	5382	2691	100.00%	-0.02	0.242	-0.4
CB-C1-17-2	310498		310498	100.00%	2.91	0.240	55.7
CB-C2-17-2	44428		44428	100.00%	0.37	0.242	7.1
CB-D1-18-2	256939	268468	262704	2.19%	2.45	0.242	46.6
CB-D2-18-2	0	0	0	ERR	-0.05	0.242	-0.9
	EXPECTED	26.4					

Chloronated Solvents Method Analytical Data Sheet

Job Name:Solvents TCE Job Num. 4154
Analyst: AC Carver Date: Jan. 2, 1989

Carbon tetrachloride Standard Calibration Curve by Linear Regress

TCE Charge (ul)	Peak Area Run 1	Run 2	Average	Predicted Charge (ul)	Percent Deviation from Actual
0.50	56001		56001	0.48	-3.04%
1.00	113359		113359	1.03	3.04%
1.50	161141		161141	1.48	-1.01%

Standard Curve Slope 105140 Intercept 5027

Desorption Coefficient: 0.82 +/- 0.04

Sample ID	Run 1 Area	Run 2 Area	Average	Percent Deviation	Detected Charge (ul)	Flow Rate (L/min)	TCE Conc. (ppm)
Run time(min):		60					
CB-13-A1-4	170891	148229	159560	7.10%	1.47	0.233	29.9
TX-13-A2-3	******		0	ERR	-0.05	0.233	-1.0
CB-14-B1-4	219036	166061	192549	13.76%	1.78	0.240	35.3
TX-14-B2-3	***	132259	66130	100.00%	0.58	0.240	11.5
CB-15-C1-4	302789	308313	305551	0.90%	2.86	0.238	57.0
TX-15-C2-3	***	177312	88656	100.00%	0.80	0.238	15.9
CB-16-D1-4	209049	233601	221325	5.55%	2.06	0.239	40.8
	EXPECTED	53.0	AVERAGE	47.6 +/-	38.3%	-23.1%	
TX-5-A1-4	260303	242149	251226	3.61%	2.34	0.232	46.4
TX-5-A2-4	23389	23233	23311	0.33%	0.17	0.232	3.4
TX-6-B1-4	250384	217936	234160	6.93%	2.18	0.234	42.8
TX-6-B2-4	72077	51062	61570	17.07%	0.54	0.234	10.6
TX-11-C1-4	172396	161971	167184	3.12%	1.54	0.235	30.2
TX-11-C2-4	134787	116517	125652	7.27%	1.15	0.235	22.5
TX-12-D1-4	186670	188432	187551	0.47%	1.74	0.238	33.5
TX-12-D2-4	93132	90699	91916	1.32%	0.83	0.238	16.0
	EXPECTED	53.0	AVERAGE	51.4 +/-	3.9%	-3.1%	

Chloronated Solvents Method Analytical Data Sheet

Job Name: Solvents TCE Job Num. 4154
Analyst: AC Carver Date: Jan. 2, 1989

Carbon tetrachloride Standard Calibration Curve by Linear Regress

TCE Charge (ul)	Peak Area Run 1	Peak Area Run 2	Peak Area Average	Predicted Charge (ul)	Percent Deviation from Actual
0.50	56001		56001	0.48	-3.04%
1.00	113359		113359	1.03	3.04%
1.50	161141		161141	1.48	-1.01%

Standard Curve Slope 105140 Intercept 5027

Desorption Coefficient: 0.82 +/- 0.04

Sample ID	Run 1 Area	Run 2 Area	Average	Percent Deviation	Detected Charge (ul)	Flow Rate (L/min)	TCE Conc. (ppm)
Run time(min):		60					
CB-13-A1-4	170891	148229	159560	7.10%	1.47	0.233	29.9
TX-13-A2-3	******		0	ERR	-0.05	0.233	-1.0
CB-14-B1-4	219036	166061	192549	13.76%	1.78	0.240	35.3
TX-14-B2-3	***	132259	66130	100.00%	0.58	0.240	11.5
CB-15-C1-4	302789	308313	305551	0.90%	2.86	0.238	57.0
TX-15-C2-3	***	177312	88656	100.00%	0.80	0.238	15.9
CB-16-D1-4	209049	233601	221325	5.55%	2.06	0.239	40.8
TX-16-D2-3	***		0	ERR	-0.05	0.239	-0.9
CB-17-E1-3	90520	116522	103521	12.56%	0.94	0.240	18.5
TX-17-E2-3	***	177312	88656	100.00%	0.80	0.240	15.7
CB-18-F1-3	122026	122026	122026	0.00%	1.11	0.232	22.8
TX-18-F2-3	***		0	ERR	-0.05	0.239	-0.9
	EXPECTED	53.0	AVERAGE	40.8 +/- 28.7%		-23.1%	
CB-A2-15-2	3566	5382	4474	20.30%	-0.01	0.242	-0.1
CB-B1-16-2	238228		238228	100.00%	2.22	0.239	42.7
CB-B2-16-2	0	5382	2691	100.00%	-0.02	0.242	-0.4
CB-C1-17-2	310498		310498	100.00%	2.91	0.240	55.7
CB-C2-17-2	44428		44428	100.00%	0.37	0.242	7.1
CB-D1-18-2	256939	268468	262704	2.19%	2.45	0.242	46.6
CB-D2-18-2	0	0	0	ERR	-0.05	0.242	-0.9
	EXPECTED	53.0	AVERAGE	ERR +/- ERR		ERR	

Chloronated Solvents Method Analytical Data Sheet

Job Name: Solvents TCE — Job Num. 4154
Analyst: AC Carver — Date: Feb 6, 1989

Carbon tetrachloride Standard Calibration Curve by Linear Regress

TCE Charge (ul)	Peak Area Run 1	Run 2	Average	Predicted Charge (ul)	Percent Deviation from Actual
0.50	77126		77126	0.49	-2.65%
1.00	155918		155918	1.03	2.65%
1.50	223085		223085	1.49	-0.88%

Standard Curve Slope 145959 Intercept 6084

Desorption Coefficient: 0.82 +/- 0.04

Sample ID	Run 1 Area	Run 2 Area	Average	Percent Deviation	Detected Charge (ul)	Flow Rate (L/min)	TCE Conc. (ppm)
Run time(min):		60					
TX-5-A1-3	208909	191043	199976	4.47%	1.33	0.232	27.2
TX-5-A2-3	2271		2271	100.00%	-0.03	0.232	-0.5
TX-6-B1-3	244023	238668	241346	1.11%	1.61	0.234	32.7
TX-6-B2-3	1759		1759	100.00%	-0.03	0.234	-0.6
TX-11-C1-3	123963	117184	120574	2.81%	0.78	0.235	15.8
TX-11-C2-3	94036	73601	83819	12.19%	0.53	0.235	10.8
TX-12-D1-3	123875		123875	100.00%	0.81	0.238	16.1
TX-12-D2-3	98409	83011	90710	8.49%	0.58	0.238	11.6
	EXPECTED	26.4	AVERAGE	28.5 +/- 9.8%		8.2%	
CB-13-A1-3	186102	183959	185031	0.58%	1.23	0.233	24.2
CB-13-A2-3	0		0	ERR	-0.04	0.233	
CB-14-B1-3	201832		201832	100.00%	1.34	0.240	25.7
CB-14-B2-3	0		0	ERR	-0.04	0.240	
CB-15-C1-3	185870		185870	100.00%	1.23	0.238	23.8
CB-15-C2-3	0		0	ERR	-0.04	0.238	
CB-16-D1-3	188612	129224	158918	18.69%	1.05	0.239	20.1
CB-16-D2-3	0					0.239	
CB-17-E1-3	173420		173420	100.00%	1.15	0.239	22.1
CB-17-E2-3	0						
CB-18-F1-3	178231		178231	100.00%	1.18	0.239	22.7
CB-18-F2-3	0						
	EXPECTED	26.4	AVERAGE	23.1 +/- 8.3%		-12.4%	

Chloronated Solvents Method Analytical Data Sheet

Job Name: Solvents TCE — Job Num. 4154
Analyst: AC Carver — Date: Feb 6, 1989

Carbon tetrachloride Standard Calibration Curve by Linear Regress

TCE Charge (ul)	Peak Area Run 1	Peak Area Run 2	Peak Area Average	Predicted Charge (ul)	Percent Deviation from Actual
0.50	77126		77126	0.49	-2.65%
1.00	155918		155918	1.03	2.65%
1.50	223085		223085	1.49	-0.88%

Standard Curve Slope 145959 Intercept 6084

Desorption Coefficient: 0.82 +/- 0.04

Sample ID	Run 1 Area	Run 2 Area	Average	Percent Deviation	Detected Charge (ul)	Flow Rate (L/min)	TCE Conc. (ppm)
Run time(min):		60					
TX-5-A1-5	176952	166736	171844	2.97%	1.14	0.232	23.2
TX-5-A2-5	6470	5739	6105	5.99%	0.00	0.232	0.0
TX-6-B1-5	169685	151172	160429	5.77%	1.06	0.234	21.4
TX-6-B2-5	2924	2447	2686	8.88%	-0.02	0.234	
TX-11-C1-5	155792	155225	155509	0.18%	1.02	0.235	20.7
TX-11-C2-5	47398	47765	47582	0.39%	0.28	0.235	5.7
TX-12-D1-5	200962	183657	192310	4.50%	1.28	0.238	25.4
TX-12-D2-5	10567		10567	100.00%	0.03	0.238	0.6
TX-19-E1-5	206514	185860	196187	5.26%	1.30	0.229	27.0
TX-19-E2-5	971		971	100.00%	-0.04	0.229	
TX-20-F1-5	203883	193029	198456	2.73%	1.32	0.230	27.2
TX-20-F2-5	11976		11976	100.00%	0.04	0.230	0.8
	EXPECTED	26.5	AVERAGE	25.4 +/- 9.9%		-4.3%	
CB-13-A1-5	156316	145945	151131	3.43%	0.99	0.233	19.6
CB-13-A2-5	35333		35333	100.00%	0.20	0.233	4.0
CB-14-B1-5	158798	155873	157336	0.93%	1.04	0.240	19.9
CB-14-B2-5	5459	5459	5459	0.00%	0.00	0.240	
CB-15-C1-5	182615	173297	177956	2.62%	1.18	0.238	22.8
CB-15-C2-5	2815	2815	2815	0.00%	-0.02	0.238	
CB-16-D1-5	141704	129224	135464	4.61%	0.89	0.239	17.1
CB-16-D2-5	0					0.239	
	EXPECTED	26.5	AVERAGE	20.8 +/- 14.3%		-21.5%	

Chloronated Solvents Method Analytical Data Sheet

Job Name: Solvents PCE — Job Num. 4154
Analyst: AC Carver — Date: Dec. 30, 1988

Perchloroethylene Standard Calibration Curve by Linear Regression

PCE Charge (ppm)	Peak Area Run 1	Peak Area Run 2	Peak Area Average	Predicted Charge (ul)	Percent Deviation from Actual
10.01	13476	12931	13204	10.14	1.25%
17.53	21595	22537	22066	17.38	-0.88%
50.10	67816	56500	62158	50.13	0.06%

Standard Curve — Slope 1224 Intercept 797

Desorption Coefficient: 1.0 +/- 0.04 (TX) 0.97 +/-0.04 (CB)

Sample ID	Run 1 Area	Run 2 Area	Average	Percent Deviation	Detected Charge (PPM)	Flow Rate (L/min)	PERC Conc. (ppm)
Run time:	40						
SB-1-3	25155	25677	25416	1.03%	20.1	0.039	20.1
SB-2-3	21409	21409	21409	0.00%	16.8	0.038	16.8
SB-3-3	25702	25702	25702	0.00%	20.3	0.037	20.3
SB-4-3	17765	17765	17765	0.00%	13.9	0.039	13.9
LB-7-3	26245	29992	28119	6.66%	22.3	0.239	22.3
LB-8-3	10117	12245	11181	9.52%	8.5	0.239	8.5
LB-9-3	40584	37827	39206	3.52%	31.4	0.242	31.4
LB-10-3	26387	25702	26045	1.32%	20.6	0.242	20.6
	EXPECTED	23.4	AVERAGE	20.8 +/-	28.1%	-11.1%	
	******DOES NOT INCLUDE LB-8-3******						
audit #AAL	9899	9643	9771	1.31%	7.3	0.239	
NBS	11210	12450	11830	5.24%	9.0	0.242	

Chloronated Solvents Method Analytical Data Sheet

Job Name: Solvents TCE — Job Num. 4154
Analyst: AC Carver — Date: Jan 31, 1989

trichloroethylene Standard Calibration Curve by Linear Regression

TCE Charge (ppm)	Peak Area Run 1	Peak Area Run 2	Peak Area Average	Predicted Charge (ppm)	Percent Deviation from Actual
10.03	11134	11419	11277	***	-0.99%
17.52	18540	18630	18585	***	0.70%
50.10	49761	48886	49324	***	-0.05%

Standard Curve Slope 948 Intercept 1865

Desorption Coefficient: 0.82 +/- 0.04

Sample ID	Run 1 Area	Run 2 Area	Average	Percent Deviation	Detected Charge (PPM)	Flow Rate (L/min)	TCE Conc. (ppm)
Run time(min):		40					
SB-1-3	23998	22990	23494	2.15%	22.82	0.039	22.8
SB-2-3	21357	17764	19561	9.18%	18.67	0.038	18.7
SB-3-3	20351	19474	19913	2.20%	19.04	0.037	19.0
SB-4-3	18546	16635	17591	5.43%	16.59	0.039	16.6
LB-7-3	25726	27516	26621	3.36%	26.12	0.239	26.1
LB-8-3	4384	4247	4316	1.59%	2.59	0.239	2.6
LB-9-3	33599	35079	34339	2.15%	34.27	0.239	34.3
LB-10-3	25057	24063	24560	2.02%	23.95	0.239	23.9
	EXPECTED	26.4	AVERAGE	23.1 +/-	27.6%	-12.5%	
	******DOES NOT INCLUDE LB-8-3******						
audit #19	10098	11508	10803	6.53%	9.43	0.240	320.6

Chloronated Solvents Method Analytical Data Sheet

Job Name: Solvents PCE Job Num. 4154
Analyst: SCS/WGD Date: Jan. 2, 1989

Perchloroethylene Standard Calibration Curve by Linear Regression

PCE Charge (ul)	Peak Area Run 1	Peak Area Run 2	Peak Area Average	Predicted Charge (ul)	Percent Deviation from Actual
0.50	51009		51009	0.50	-0.92%
1.00	100682	100756	100719	1.01	0.92%
1.50	147752		147752	1.50	-0.31%

Standard Curve Slope 96743 Intercept 3084

Desorption Coefficient: 1.0 +/- 0.04 (TX) 0.97 +/-0.04 (CB)

Sample ID	Run 1 Area	Run 2 Area	Average	Percent Deviation	Detected Charge (ul)	Flow Rate (L/min)	PERC Conc. (ppm)
Run time:	60						
CB-13-A1-4	115236	120574	117905	2.26%	1.2	0.233	20.0
TX-13-A2-4	***	2737	1369	100.00%	0.0	0.233	-0.3
CB-14-B1-4	180764	138953	159859	13.08%	1.6	0.240	26.5
TX-14-B2-3	***	**	0	ERR	0.0	0.240	-0.5
CB-15-C1-4	259637	245400	252519	2.82%	2.6	0.238	42.5
TX-15-C2-3	****		0	ERR	0.0	0.238	-0.5
CB-16-D1-4	148761	169549	159155	6.53%	1.6	0.239	26.5
TX-16-D2-3							
	EXPECTED	47.0	AVERAGE	28.9 +/-	33.2%	-38.6%	
TX-5-A1-4	182812	175320	179066	2.09%	1.82	0.232	31.7
TX-5-A2-4	27170	24944	26057	4.27%	0.24	0.232	4.1
TX-6-B1-4	162946	147962	155454	4.82%	1.58	0.234	27.2
TX-6-B2-4	59799	59856	59828	0.05%	0.59	0.234	10.1
TX-11-C1-4	93413	77150	85282	9.53%	0.85	0.235	14.6
TX-11-C2-4	167706	143395	155551	7.81%	1.58	0.235	27.1
TX-12-D1-4	107002	114454	110728	3.37%	1.11	0.238	18.9
TX-12-D2-4	120314	122413	121364	0.86%	1.22	0.238	20.8
	EXPECTED	78.3	AVERAGE	23.1 +/-	33.6%	-70.5%	

Chloronated Solvents Method Analytical Data Sheet

Job Name: Solvents PCE — Job Num. 4154
Analyst: SCS/WGD — Date: Jan 2, 1989

Perchloroethylene Standard Calibration Curve by Linear Regression

PCE Charge (ul)	Peak Area Run 1	Peak Area Run 2	Peak Area Average	Predicted Charge (ul)	Percent Deviation from Actual
0.50	51009		51009	0.50	-0.92%
1.00	100682	100756	100719	1.01	0.92%
1.50	147752		147752	1.50	-0.31%

Standard Curve — Slope 96743 Intercept 3084

Desorption Coefficient: 1.0 +/- 0.04 (TX) 0.97 +/-0.04 (CB)

Sample ID	Run 1 Area	Run 2 Area	Average	Percent Deviation	Detected Charge (ul)	Flow Rate (L/min)	PERC Conc. (ppm)
Run time:	60						
CB-13-A1-3	160067	115960	138014	15.98%	1.4	0.233	23.5
TX-13-A2-3	2737	2737	2737	0.00%	0.0	0.233	-0.1
CB-14-B1-3	135649	103298	119474	13.54%	1.2	0.240	19.7
TX-14-B2-3	***	**	0	ERR	0.0	0.240	-0.5
CB-15-C1-3	126749	121041	123895	2.30%	1.2	0.238	20.6
TX-15-C2-3	****		0	ERR	0.0	0.238	-0.5
CB-16-D1-3	109429		109429	100.00%	1.1	0.239	18.0
TX-16-D2-3							
CB-17-E1-3	80335	80335	80335	0.00%	0.8	0.240	13.1
TX-17-E2-3							
CB-18-F1-3	84095	84095	84095	0.00%	0.8	0.232	14.2
TX-18-F2-3							
	EXPECTED	23.4	AVERAGE	20.4 +/- 11.2%		-12.6%	
	*****DOES NOT INCLUDE CB-17-E1-3, CB-18-F1-3*****						
CB-A1-15-2	76724	65719	71222	7.73%	0.70	0.240	11.9
CB-A2-15-2	3566	5382	4474	20.30%	0.01	0.242	0.2
CB-B1-16-2	163498		163498	100.00%	1.66	0.239	28.1
CB-B2-16-2		5382	5382	0.00%	0.02	0.239	0.4
CB-C1-17-2	198412	231054	214733	7.60%	2.19	0.240	36.9
CB-C2-17-2	13500		13500	100.00%	0.11	0.240	1.8
CB-D1-18-2	164760	165444	165102	0.21%	1.67	0.230	29.4
CB-D2-18-2	0	0	0	ERR	-0.03	0.242	-0.5
	EXPECTED	23.4	AVERAGE	26.6 +/- 39.6%		13.5%	

Chloronated Solvents Method Analytical Data Sheet

Job Name: Solvents PCE Job Num. 4154
Analyst: ACC Date: Feb 6, 1989

Perchloroethylene Standard Calibration Curve by Linear Regression

PCE Charge (ul)	Peak Area Run 1	Run 2	Average	Predicted Charge (ul)	Percent Deviation from Actual
0.50	70669	74220	72445	0.48	-3.97%
1.00	148707		148707	1.04	3.97%
1.50	208721		208721	1.48	-1.32%

Standard Curve Slope 136277 Intercept 7014

Desorption Coefficient: 1.0 +/- 0.04 (TX) 0.97 +/-0.04 (CB)

Sample ID	Run 1 Area	Run 2 Area	Average	Percent Deviation	Detected Charge (ul)	Flow Rate (L/min)	PERC Conc. (ppm)
Run time:	60						
TX-5-A1-3	201983	184422	193203	4.54%	1.4	0.232	23.1
TX-5-A2-3	3297		3297	100.00%	0.0	0.232	-0.5
TX-6-B1-3	243745	230997	237371	2.69%	1.7	0.234	28.3
TX-6-B2-3	624		624	100.00%	0.0	0.234	-0.8
TX-11-C1-3	72673	72183	72428	0.34%	0.5	0.235	8.0
TX-11-C2-3	146520	112890	129705	12.96%	0.9	0.235	15.0
TX-12-D1-3	62247		62247	100.00%	0.4	0.238	6.7
TX-12-D2-3	151009	127647	139328	8.38%	1.0	0.238	16.0
	EXPECTED	23.4	AVERAGE	24.3 +/- 11.1%		3.9%	
CB-13-A1-3	187419	183503	185461	1.06%	1.31	0.233	22.7
CB-13-A2-3	0	0	0	ERR	-0.05	0.233	-0.9
CB-14-B1-3	194520		194520	100.00%	1.38	0.240	23.2
CB-14-B2-3	5774		5774	100.00%	-0.01	0.240	-0.2
CB-15-C1-3	187622		187622	100.00%	1.33	0.238	22.5
CB-15-C2-3	0		0	ERR	-0.05	0.238	-0.9
CB-16-D1-3	177022		177022	100.00%	1.25	0.239	21.1
CB-16-D2-3	0		0	ERR	-0.05	0.239	-0.9
CB-17-E1-3	119153		119153	100.00%	0.82	0.239	13.9
CB-17-E2-3	0						
CB-18-F1-3	120058		120058	100.00%	0.83	0.239	14.0
	0						
	EXPECTED	23.5	AVERAGE	22.4 +/- 4.0%		-4.7%	

Does not include CB-17-E1-3 and CB-18-F1-3
(Carboxen was not preconditioned)